PATENT ABSTRACTS OF JAPAN

(11)Publication number : 08-259279
(43)Date of publication of application : 08.10.1996

(51)Int.CI. C03C 27/12
B32B 17/10
B60J 1/00
E06B 5/00

(21)Application number : 07-165489
(22)Date of filing : 30.06.1995

(71)Applicant : CENTRAL GLASS CO LTD
(72)Inventor : KONDO TAKESHI

(30)Priority
Priority number : 07 7944 Priority date : 23.01.1995 Priority country : JP

(54) LAMINATED GLASS AND ITS PRODUCTION

(57)Abstract:
PURPOSE: To impart satisfactory heat insulating performance, UV shielding performance, etc., at the time of interposing an intermediate film layer between two transparent glasses to form the laminated glass by dispersing functional superfine particles with the diameter specified or below in the intermediate film layer.

CONSTITUTION: Functional superfine particles (e.g. conductive antimony-contg. tin oxide superfine particles) having ≤0.2μm diameter are dispersed in an intermediate film. A polyvinyl butyral resin film is preferably used as the intermediate film. When the functional superfine particles are dispersed, the particles having ≤0.2μm diameter are uniformly dispersed in a solvent dissolving the polyvinyl butyral resin, and then the solvent is uniformly dissolved in the resin along with an additive such as a plasticizer and kneaded, formed into film and dried. Subsequently, at least two transparent glass sheets are laminated by using the obtained intermediate film and vitrified, and a laminated glass is produced.

LEGAL STATUS

[Date of request for examination]	24.02.1998
[Date of sending the examiner's decision of rejection]	
[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]	
[Date of final disposal for application]	
[Patent number]	3154645
[Date of registration]	02.02.2001
[Number of appeal against examiner's decision of rejection]	
[Date of requesting appeal against examiner's decision of rejection]	

* NOTICES *

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

CLAIMS

[Claim(s)]
[Claim 1] In the laminated glass which has an interlayer layer between the transparence glass plate-like objects of at least two sheets, particle size in this interlayer layer Laminated glass characterized by making it come to distribute a functional ultrafine particle 0.2 micrometers or less.
[Claim 2] Laminated glass according to claim 1 with which said interlayer is characterized by being the polyvinyl-butyral system resin film.
[Claim 3] Laminated glass according to claim 1 with which said interlayer is characterized by being the ethylene-vinylacetate copolymer system resin film.
[Claim 4] the particle size of said functional ultrafine particle -- 0.15 to 0.001 micrometer it is -- laminated glass according to claim 1 to 3 characterized by things.
[Claim 5] Laminated glass according to claim 1 to 4 with which the mixed rate of said functional ultrafine particle is characterized by being less than [10.0wt%].
[Claim 6] Laminated glass according to claim 1 to 5 with which the mixed rate of said functional ultrafine particle is characterized by being 2.0 - 0.01wt%.
[Claim 7] Said functional ultrafine particle Sn, Ti, Si, Zn, Zr, Fe, aluminum, Cr, Co, Ce, In, nickel, Ag, Cu, Pt, Mn, Ta, and W, The metal of V and Mo, an oxide, a nitride, a sulfide, or Sb and F Each independent object of a dope object, Or laminated glass according to claim 1 to 6 characterized by being the coat object which covered the composite which comes to choose at least two or more sorts from these, the mixture which contains an organic resin object in each independent object or a composite concerned further, or an organic resin object.
[Claim 8] Laminated glass according to claim 1 to 7 with which said interlayer is characterized by coming to contain each independent one of an organic system ultraviolet ray absorbent, an organic system heat ray absorbent, or a pigment, or these.
[Claim 9] Laminated glass according to claim 1 to 8 with which said laminated glass is characterized by being structural glass.
[Claim 10] Laminated glass according to claim 1 to 8 with which said laminated glass is characterized by being window glass for automobiles.
[Claim 11] Particle size in the approach of manufacturing the laminated glass which has an interlayer layer between the transparence glass plate-like objects of at least two sheets The manufacture approach of the laminated glass characterized by carrying out laminated-glass-ized processing for said glass plate of at least two sheets using this interlayer that made the functional ultrafine particle 0.2 micrometers or less distribute.
[Claim 12] The manufacture approach of laminated glass according to claim 11 that said interlayer is characterized by being the polyvinyl-butyral system resin film.
[Claim 13] said polyvinyl-butyral system resin film -- particle size 0.2 micrometers Make it distribute and it considers as a functional ultrafine particle distribution plasticizer. the following functional ultrafine particles -- the inside of a plasticizer -- less than [80.0wt%] -- This functional ultrafine particle distribution plasticizer subsequently, in polyvinyl-butyral system resin Distributed addition is carried out at least. polyvinyl-butyral system resin -- receiving -- a functional ultrafine particle distribution plasticizer -- less than [50wt%] -- The manufacture approach of the laminated glass according to claim 11 to 12 characterized by having added other additives suitably and obtaining a functional ultrafine particle from the raw material resin for film distributed to homogeneity by carrying out mixed kneading.
[Claim 14] said functional ultrafine particle distribution plasticizer -- particle size 0.2 micrometers Following

0.001 the functional ultrafine particle more than mum -- the inside of a plasticizer -- less than [20.0wt%] -- the manufacture approach of the laminated glass according to claim 12 to 13 characterized by being the thing which it makes it come to distribute.
[Claim 15] Said particle size to the solvent which dissolves polyvinyl-butyral system resin 0.2 micrometers Following 0.001 After distributing the functional ultrafine particle more than mum homogeneity or in the shape of homogeneity at least, [said interlayer] Suitably, with a plasticizer and other additives, make polyvinyl-butyral system resin carry out the homogeneity dissolution, carry out mixed kneading, and the solvent concerned is film-ized from the raw material resin for film. The manufacture approach of the laminated glass according to claim 11 characterized by being the polyvinyl-butyral system resin film dried and obtained by 50 - 110 **.
[Claim 16] Said particle size at least to the polyvinyl-butyral system resin which said interlayer heated beyond the temperature of 55-90 degrees C which is a glass transition point, and softened 0.2 micrometers Following 0.001 The manufacture approach of the laminated glass according to claim 11 characterized by being the polyvinyl-butyral system resin film which added the functional ultrafine particle more than mum directly, and carried out mixed kneading, and which was obtained from the raw material resin for film which carried out homogeneity distribution.
[Claim 17] The manufacture approach of laminated glass according to claim 11 that said interlayer is characterized by being ethylene-vinylacetate copolymer system resin.
[Claim 18] said ethylene-vinylacetate copolymer system resin -- particle size 0.2 micrometers Make it distribute and it considers as a functional ultrafine particle distribution plasticizer. the following functional ultrafine particles -- the inside of a plasticizer -- less than [80.0wt%] -- This functional ultrafine particle distribution plasticizer subsequently, in ethylene-vinylacetate copolymer system resin It adds at least. ethylene-vinylacetate copolymer system resin -- receiving -- a functional ultrafine particle distribution plasticizer -- less than [50wt%] -- Claim 11 characterized by having added other additives suitably and obtaining a functional ultrafine particle from the raw material resin for film distributed to homogeneity by carrying out mixed kneading, and the manufacture approach of laminated glass given in 17.
[Claim 19] said functional ultrafine particle distribution plasticizer -- particle size 0.2 micrometers Following 0.001 mum the above functional ultrafine particle -- the inside of a plasticizer -- less than [20.0wt%] -- the manufacture approach of laminated glass claims 11 and 17 characterized by being the thing which it makes it come to distribute thru/or given in 18.
[Claim 20] Said particle size to the solvent for ethylene-vinylacetate copolymer system resin 0.2 micrometers Following 0.001 After distributing the functional ultrafine particle more than mum homogeneity or in the shape of homogeneity at least, [said interlayer] Suitably, with other additives, make ethylene-vinylacetate copolymer system resin carry out the homogeneity dissolution, carry out mixed kneading, and the solvent concerned is film-ized from the raw material resin for film. Claim 11 characterized by being ethylene-vinylacetate copolymer system resin dried and obtained by 50 - 110 **, and the manufacture approach of laminated glass given in 17.
[Claim 21] Said particle size at least to the ethylene-vinylacetate copolymer system resin which said interlayer heated beyond the temperature of 55-90 degrees C which is a glass transition point , and softened 0.2 micrometers Following 0.001 Claim 11 characterize by be ethylene-vinylacetate copolymer system resin which added the functional ultrafine particle more than mum directly , and carried out mixed kneading , and which was obtained from the raw material resin for film which carried out homogeneity distribution , and the manufacture approach of laminated glass given in 17 .
[Claim 22] Said functional ultrafine particle Sn, Ti, Si, Zn, Zr, Fe, aluminum, Cr, Co, Ce, In, nickel, Ag, Cu, Pt, Mn, Ta, and W, The metal of V and Mo, an oxide, a nitride, a sulfide, or Sb and F Each independent object of a dope object, Or the manufacture approach of the laminated glass according to claim 11 to 21 characterized by being the coat object which covered the composite which comes to choose at least two or more sorts from these, the mixture which contains an organic resin object in each independent object or a composite concerned further, or an organic resin object.
[Claim 23] The manufacture approach of laminated glass according to claim 11 to 17 that film-ization of said raw material resin for film is characterized by being based on the mold extrusion method or the calendering roll method of a conventional method.
[Claim 24] The manufacture approach of laminated glass according to claim 11 to 17 that said laminated-glass-ized processing is characterized by being based on the autoclave method.

[Claim 25] Claims 11 and 18 characterized by basing said laminated-glass-ized processing on heating for 20 - 30 minutes in the temperature requirement of 80 - 120 ** while carrying out a temperature up from ordinary temperature to 120 ** under reduced pressure thru/or the manufacture approach of laminated glass given in 22.

[Translation done.]

* NOTICES *

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

CLAIMS

[Claim(s)]
[Claim 1] In the laminated glass which has an interlayer layer between the transparence glass plate-like objects of at least two sheets, particle size in this interlayer layer Laminated glass characterized by making it come to distribute a functional ultrafine particle 0.2 micrometers or less.
[Claim 2] Laminated glass according to claim 1 with which said interlayer is characterized by being the polyvinyl-butyral system resin film.
[Claim 3] Laminated glass according to claim 1 with which said interlayer is characterized by being the ethylene-vinylacetate copolymer system resin film.
[Claim 4] the particle size of said functional ultrafine particle -- 0.15 to 0.001 micrometer it is -- laminated glass according to claim 1 to 3 characterized by things.
[Claim 5] Laminated glass according to claim 1 to 4 with which the mixed rate of said functional ultrafine particle is characterized by being less than [10.0wt%].
[Claim 6] Laminated glass according to claim 1 to 5 with which the mixed rate of said functional ultrafine particle is characterized by being 2.0 - 0.01wt%.
[Claim 7] Said functional ultrafine particle Sn, Ti, Si, Zn, Zr, Fe, aluminum, Cr, Co, Ce, In, nickel, Ag, Cu, Pt, Mn, Ta, and W, The metal of V and Mo, an oxide, a nitride, a sulfide, or Sb and F Each independent object of a dope object, Or laminated glass according to claim 1 to 6 characterized by being the coat object which covered the composite which comes to choose at least two or more sorts from these, the mixture which contains an organic resin object in each independent object or a composite concerned further, or an organic resin object.
[Claim 8] Laminated glass according to claim 1 to 7 with which said interlayer is characterized by coming to contain each independent one of an organic system ultraviolet ray absorbent, an organic system heat ray absorbent, or a pigment, or these.
[Claim 9] Laminated glass according to claim 1 to 8 with which said laminated glass is characterized by being structural glass.
[Claim 10] Laminated glass according to claim 1 to 8 with which said laminated glass is characterized by being window glass for automobiles.
[Claim 11] Particle size in the approach of manufacturing the laminated glass which has an interlayer layer between the transparence glass plate-like objects of at least two sheets The manufacture approach of the laminated glass characterized by carrying out laminated-glass-ized processing for said glass plate of at least two sheets using this interlayer that made the functional ultrafine particle 0.2 micrometers or less distribute.
[Claim 12] The manufacture approach of laminated glass according to claim 11 that said interlayer is characterized by being the polyvinyl-butyral system resin film.
[Claim 13] said polyvinyl-butyral system resin film -- particle size 0.2 micrometers Make it distribute and it considers as a functional ultrafine particle distribution plasticizer. the following functional ultrafine particles -- the inside of a plasticizer -- less than [80.0wt%] -- This functional ultrafine particle distribution plasticizer subsequently, in polyvinyl-butyral system resin Distributed addition is carried out at least. polyvinyl-butyral system resin -- receiving -- a functional ultrafine particle distribution plasticizer -- less than [50wt%] -- The manufacture approach of the laminated glass according to claim 11 to 12 characterized by having added other additives suitably and obtaining a functional ultrafine particle from the raw material resin for film distributed to homogeneity by carrying out mixed kneading.
[Claim 14] said functional ultrafine particle distribution plasticizer -- particle size 0.2 micrometers Following

0.001 the functional ultrafine particle more than mum -- the inside of a plasticizer -- less than [20.0wt%] -- the manufacture approach of the laminated glass according to claim 12 to 13 characterized by being the thing which it makes it come to distribute.

[Claim 15] Said particle size to the solvent which dissolves polyvinyl-butyral system resin 0.2 micrometers Following 0.001 After distributing the functional ultrafine particle more than mum homogeneity or in the shape of homogeneity at least, [said interlayer] Suitably, with a plasticizer and other additives, make polyvinyl-butyral system resin carry out the homogeneity dissolution, carry out mixed kneading, and the solvent concerned is film-ized from the raw material resin for film. The manufacture approach of the laminated glass according to claim 11 characterized by being the polyvinyl-butyral system resin film dried and obtained by 50 - 110 **.

[Claim 16] Said particle size at least to the polyvinyl-butyral system resin which said interlayer heated beyond the temperature of 55-90 degrees C which is a glass transition point, and softened 0.2 micrometers Following 0.001 The manufacture approach of the laminated glass according to claim 11 characterized by being the polyvinyl-butyral system resin film which added the functional ultrafine particle more than mum directly, and carried out mixed kneading, and which was obtained from the raw material resin for film which carried out homogeneity distribution.

[Claim 17] The manufacture approach of laminated glass according to claim 11 that said interlayer is characterized by being ethylene-vinylacetate copolymer system resin.

[Claim 18] said ethylene-vinylacetate copolymer system resin -- particle size 0.2 micrometers Make it distribute and it considers as a functional ultrafine particle distribution plasticizer. the following functional ultrafine particles -- the inside of a plasticizer -- less than [80.0wt%] -- This functional ultrafine particle distribution plasticizer subsequently, in ethylene-vinylacetate copolymer system resin It adds at least. ethylene-vinylacetate copolymer system resin -- receiving -- a functional ultrafine particle distribution plasticizer -- less than [50wt%] -- Claim 11 characterized by having added other additives suitably and obtaining a functional ultrafine particle from the raw material resin for film distributed to homogeneity by carrying out mixed kneading, and the manufacture approach of laminated glass given in 17.

[Claim 19] said functional ultrafine particle distribution plasticizer -- particle size 0.2 micrometers Following 0.001 mum the above functional ultrafine particle -- the inside of a plasticizer -- less than [20.0wt%] -- the manufacture approach of laminated glass claims 11 and 17 characterized by being the thing which it makes it come to distribute thru/or given in 18.

[Claim 20] Said particle size to the solvent for ethylene-vinylacetate copolymer system resin 0.2 micrometers Following 0.001 After distributing the functional ultrafine particle more than mum homogeneity or in the shape of homogeneity at least, [said interlayer] Suitably, with other additives, make ethylene-vinylacetate copolymer system resin carry out the homogeneity dissolution, carry out mixed kneading, and the solvent concerned is film-ized from the raw material resin for film. Claim 11 characterized by being ethylene-vinylacetate copolymer system resin dried and obtained by 50 - 110 **, and the manufacture approach of laminated glass given in 17.

[Claim 21] Said particle size at least to the ethylene-vinylacetate copolymer system resin which said interlayer heated beyond the temperature of 55-90 degrees C which is a glass transition point , and softened 0.2 micrometers Following 0.001 Claim 11 characterize by be ethylene-vinylacetate copolymer system resin which added the functional ultrafine particle more than mum directly , and carried out mixed kneading , and which was obtained from the raw material resin for film which carried out homogeneity distribution , and the manufacture approach of laminated glass given in 17 .

[Claim 22] Said functional ultrafine particle Sn, Ti, Si, Zn, Zr, Fe, aluminum, Cr, Co, Ce, In, nickel, Ag, Cu, Pt, Mn, Ta, and W, The metal of V and Mo, an oxide, a nitride, a sulfide, or Sb and F Each independent object of a dope object, Or the manufacture approach of the laminated glass according to claim 11 to 21 characterized by being the coat object which covered the composite which comes to choose at least two or more sorts from these, the mixture which contains an organic resin object in each independent object or a composite concerned further, or an organic resin object.

[Claim 23] The manufacture approach of laminated glass according to claim 11 to 17 that film-ization of said raw material resin for film is characterized by being based on the mold extrusion method or the calendering roll method of a conventional method.

[Claim 24] The manufacture approach of laminated glass according to claim 11 to 17 that said laminated-glass-ized processing is characterized by being based on the autoclave method.

[Claim 25] Claims 11 and 1[redacted]haracterized by basing said laminated-glass-[redacted]ed processing on heating for 20 - 30 minutes in the temperature requirement of 80 - 120 ** while carrying out a temperature up from ordinary temperature to 120 ** under reduced pressure thru/or the manufacture approach of laminated glass given in 22.

[Translation done.]

* NOTICES *

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Industrial Application] This invention relates to the laminated glass which becomes by doubling and processing various kinds of functional ultrafine particles, such as coloring, a heat ray, and ultraviolet-rays cutoff film, electric-wave transparency, using the resin interlayer layer which it has suitably, and its manufacture approach.
[0002] While having the ultraviolet-rays cutoff which becomes kind to solar radiation permeability, a great environment, and a great man who make the air conditioning effectiveness improve It is what has visible-ray permeability broad from a comparatively high thing to a low thing. Reduction can do electromagnetic interferences, such as radio disturbance in broadcast of AM electric wave, FM electric wave, etc., or a ghost phenomenon. It is electric-wave transparency mold heat ray ultraviolet-rays electric shielding glass usable as laminated glass of colored [colorlessness to], and various color tones which needs electric-wave permeability ability. The aperture material for automobiles, for example, a front window, of course especially as structural aperture material A rear window, a side window, or a sunroof is provided with the aperture material for airplanes, the laminated glass which, further in addition to this, has broadly applicable useful functionality, such as an industrial member, and its manufacture approach again.
[0003]
[Description of the Prior Art] functional grant of the clearance in recent years and structural glass, coloring and heat insulation, ultraviolet-rays cutoff, electric-wave transparency, etc., etc. -- of course -- the glass for vehicles -- also setting -- in the car -- a connoisseur -- the solar-radiation energy which carries out ON is covered, and from the purpose which reduces a temperature rise in the car and a cooling load, further, human material both sides and in order to make it environment-friendly, heat ray electric shielding glass and the thing which added ultraviolet-rays electric shielding are adopted as vehicles. Moreover, the following is known as what the high permeability ability of various electric waves is required with the high temperature line ultraviolet-rays electric shielding engine performance, and distributed the particle or the ultrafine particle to the interlayer of laminated glass especially, having light permeability sufficient in the Green color tone in this glass for vehicles recently especially.
[0004] For example, at least one sort of light absorption agents carried from the group which consists of a benzotriazol derivative expressed with the general formula which the interlayer for short wave Nagamitsu line cutoff nature laminated glass is indicated by JP,2-22152,A, and was specified as it, At least 90 % of the weight It consists of plasticization polyvinyl butyral resin containing the particle-like mineral matter of the particle size distribution in the size range which is 250-400nm. 400nm The thing which the light of the following wavelength is intercepted [thing] substantially and makes light with a wavelength of 450nm or more penetrate substantially is indicated, and the content of a light absorption agent is 0.4-6. It is weight % and the content of particle-like mineral matter It is indicated that it is 2 - 17 % of the weight.
[0005] Moreover, to JP,4-160041,A The windowpane for automobiles is indicated and it is mean particle diameter 0.1 between transparence plate-like part material. mum The thing which comes to form the mixolimnion of the following ultrafine particles and a glass component is indicated. Two ultrafine particles and a glass component are pinched between transparence plate-like part material, and transparence plate-like part material is pasted up by the glass component, Or the interlayer (PVB) of plastics is prepared between transparence plate-like part material, and it is particle size 0.1 between this interlayer and each plate-like part

material, respectively. mum It comes to form the mixolimnion of the following ultrafine particles and a glass component. Or mean particle diameter 0.3-0.5 mum Making the particle for spacers intermingled in a mixolimnion etc. is indicated.
[0006] Moreover, to JP,4-261842,A The interlayer which the glass laminate is indicated and was arranged between organic glass, the transparent body, and organic glass and the transparent body, Are the glass laminate which **** and what is formed with the resin constituent with which an interlayer contains the ethylene ethyl acrylate copolymerization resin which carried out graft denaturation of the vinylsilane is indicated. Ethylene ethyl acrylate copolymerization resin 100 with which the resin constituent carried out graft denaturation of the vinylsilane Containing the weight section, and the silicon-dioxide particle 3 - 30 weight sections is indicated.
[0007]
[Problem(s) to be Solved by the Invention] As mentioned above, the interlayer for short wave Nagamitsu line cutoff nature laminated glass indicated by JP,2-22152,A etc. At least 90 % of the weight added by polyvinyl butyral resin is 250-400nm. The particle-like mineral matter of the particle size distribution in a size range is 400nm as a light-scattering agent. So that the following ultraviolet-rays parts may be scattered It carries out, alternative absorption of a light absorption agent is promoted, and it is 400nm. While intercepting the light of the following wavelength substantially Light transmission in [wavelength] 450-700nm For example, 70 etc.% or more etc., 450nm Wavelength of 420nm which the light of the above wavelength is made to penetrate substantially, transparency is held, and there is moreover no muddiness at the observation in the edge of the white lamp image of 100W, and shows the yellow taste Although the light transmission which can be set is also 50% or more and it is said that a good adhesive property is shown For example, heat insulation property is brought about and, for a **** reason, at least 90 % of the weight is 250-400nm. When the adiathermic particle-like mineral matter of the particle size distribution in a size range is added to polyvinyl butyral resin For example, 450-700nm It is what the publication of the publication of light transmission being able to adopt now as a windowpane for automobiles moreover at 70% or more in the wavelength range to suggest does not have, either. Of course, that the particle size of adiathermic particle-like mineral matter is comparatively large needs to also make [many] the addition with 2 - 17 % of the weight.
[0008] Moreover, for example, the windowpane for automobiles indicated by JP,4-160041,A It is mean particle diameter 0.1 between transparence plate-like part material. mum A mixolimnion with the glass component of the following ultrafine particles, organic silicon, or an organic silicon compound is formed. The polyvinyl butyral (PVB) and glass which are the glass of a glass laminate or the middle class of plastics were joined. The defroster ability as an object for heaters, the infrared reflex function as an object for an air conditioning effectiveness rise, and/or sheet resistance are about 500. It will have the electromagnetic shielding function which are omega/opening. PVB Ethylene-vinylacetate copolymer system resin film (EVA) A heat insulation function in the configuration of the usual laminated glass which joined the glass of two sheets only by the interlayer etc. -- There is a factor which is just going to be apprehensive about the ability of adhesive strength equivalent to usual laminated glass to be obtained, and is raised also in cost so that an ultraviolet-rays cutoff function, an electric-wave transparency function or colorlessness thru/or coloring may not be discovered to coincidence.
[0009] Moreover, for example, the glass laminate indicated by JP,4-261842,A Ethylene ethyl acrylate copolymerization resin 100 which is for using organic glass and carried out graft denaturation of the vinylsilane As opposed to the weight section Since particle size is shorter than the wavelength (400-780nm) of a visible ray by making it contain the silicon-dioxide particles 3 which are 0.1 -400mmicro, such as colloidal silica and an ultrafine particle silica, - 30 weight sections, and making particle size into less than [400mmicro], Although dispersion of the light which passes an interlayer tends to be prevented and it is going to make a cloudy improvement of the interlayer effective Whenever [cloudy weather] (Hayes) is JIS K6714. It is 4% or less extent in the based measurement, and is hard to call it not necessarily enough windowpanes for automobiles, especially a windshield.
[0010]
[Means for Solving the Problem] It is distributing in an interlayer layer suitably and making it contain a functional ultrafine particle, without affecting the interlayer layer for laminated glass which makes this invention in view of such a conventional point, and is used from the former. Functional characteristics, such as heat insulation property, ultraviolet-rays cutoff engine performance, and electric-wave permeability ability, are

given, moreover, it glares with reservation and reflexibility of control of the color tone of a clearance thru/or coloring, and fluoroscopy nature, and prevention of admiration etc. is brought about with sufficient balance. **, Can acquire the quality which is not different from conventional laminated glass, and neither special component presentation glass nor special surface treatment glass is needed. And it remains as it is and a laminated-glass production line present in use can be performed by the laminated-glass-ized processing activity. For example, cheaply, easy moreover, it can respond to the magnitude and the gestalt of glass freely, and glass, glass and glass, plastics, bilayer glass, etc. can be manufactured. Of course, it can apply also to the glass for windshields enough especially, and structural aperture material offers the aperture material for automobiles, the aperture material for airplanes, and the useful functional laminated glass used as the optimal thing for the latest needs.
[0011] That is, in the laminated glass which has an interlayer layer between the transparence glass plate-like objects of at least two sheets, particle size this invention in this interlayer layer Laminated glass characterized by making it come to distribute a functional ultrafine particle 0.2 micrometers or less.
[0012] And laminated glass with which said interlayer is characterized by being the polyvinyl-butyral system resin film and which was mentioned above. Moreover, laminated glass with which said interlayer is characterized by being the ethylene-vinylacetate copolymer system resin film and which was mentioned above.
[0013] furthermore, the particle size of said functional ultrafine particle -- 0.15 to 0.001 micrometer it is -- laminated glass which is characterized by things and which was mentioned above. Furthermore, laminated glass with which the mixed rate of said functional ultrafine particle is characterized by being less than [10.0wt%] and which was mentioned above.
[0014] Laminated glass with which the mixed rate of said functional ultrafine particle is characterized by being 2.0 - 0.01wt% further again and which was mentioned above. Said functional ultrafine particle Furthermore, Sn, Ti, Si, Zn, Zr, Fe, aluminum, Cr, Co, Ce, In, nickel, Ag, Cu, Pt, Mn, The metal of Ta, W, V, and Mo, an oxide, a nitride, a sulfide, or Sb and F The composite which comes to choose at least two or more sorts from each independent object of a dope object, or these, Or laminated glass which is characterized by being the coat object which covered the mixture or the organic resin object which contains an organic resin object in each independent object or a composite concerned further and which was mentioned above.
[0015] Furthermore, laminated glass with which said interlayer is characterized by coming to contain each independent one of an organic system ultraviolet ray absorbent, an organic system heat ray absorbent, or a pigment, or these and which was mentioned above.
[0016] Furthermore, laminated glass with which said laminated glass is characterized by being structural glass and which was mentioned above. Furthermore, laminated glass with which said laminated glass is characterized by being window glass for automobiles and which was mentioned above.
[0017] And particle size in the approach of manufacturing the laminated glass which has an interlayer layer between the transparence glass plate-like objects of at least two sheets The manufacture approach of the laminated glass characterized by carrying out laminated-glass-ized processing for said glass plate of at least two sheets using this interlayer that made the functional ultrafine particle 0.2 micrometers or less distribute.
[0018] Moreover, the manufacture approach of the laminated glass mentioned above that said interlayer is characterized by being the polyvinyl-butyral system resin film. furthermore, said polyvinyl-butyral system resin film -- particle size 0.2 micrometers Make it distribute and it considers as a functional ultrafine particle distribution plasticizer. the following functional ultrafine particles -- the inside of a plasticizer -- less than [80.0wt%] -- This functional ultrafine particle distribution plasticizer subsequently, in polyvinyl-butyral system resin polyvinyl-butyral system resin -- receiving -- a functional ultrafine particle distribution plasticizer -- less than [50wt%] -- the manufacture approach of the laminated glass which carries out distributed addition at least, adds other additives suitably and is characterized by obtaining a functional ultrafine particle from the raw material resin for film distributed to homogeneity by carrying out mixed kneading and which was mentioned above.
[0019] furthermore, said functional ultrafine particle distribution plasticizer -- particle size 0.2 micrometers Following 0.001 the functional ultrafine particle more than mum -- the inside of a plasticizer -- less than [20.0wt%] -- the manufacture approach of the laminated glass which is characterized by being the thing which it makes it come to distribute and which was mentioned above.
[0020] Moreover, said particle size to the solvent which dissolves polyvinyl-butyral system resin 0.2 micrometers Following 0.001 After distributing the functional ultrafine particle more than mum homogeneity or

in the shape of homogeneity at least, [said interlayer] Suitably, with a plasticizer and other additives, make polyvinyl-butyral system resin carry out the homogeneity dissolution, carry out mixed kneading, and the solvent concerned is film-ized from the raw material resin for film. The manufacture approach of the laminated glass which is characterized by being the polyvinyl-butyral system resin film dried and obtained by 50 - 110 ** and which was mentioned above.

[0021] Moreover, said particle size at least to the polyvinyl-butyral system resin which said interlayer heated beyond the temperature of 55-90 degrees C which is a glass transition point, and softened 0.2 micrometers Following 0.001 The manufacture approach of the laminated glass which is characterized by being the polyvinyl-butyral system resin film which added the functional ultrafine particle more than mum directly, and carried out mixed kneading, and which was obtained from the raw material resin for film which carried out homogeneity distribution and which was mentioned above.

[0022] Moreover, the manufacture approach of the laminated glass mentioned above that said interlayer is characterized by being ethylene-vinylacetate copolymer system resin. furthermore, said ethylene-vinylacetate copolymer system resin -- particle size 0.2 micrometers Make it distribute and it considers as a functional ultrafine particle distribution plasticizer. the following functional ultrafine particles -- the inside of a plasticizer solution -- less than [80.0wt%] -- This functional ultrafine particle distribution plasticizer subsequently, in ethylene-vinylacetate copolymer system resin ethylene-vinylacetate copolymer system resin -- receiving -- a functional ultrafine particle distribution plasticizer -- less than [50wt%] -- the manufacture approach of the laminated glass which is characterized by having added at least, having added other additives suitably, and obtaining a functional ultrafine particle from the raw material resin for film distributed to homogeneity by carrying out mixed kneading and which was mentioned above.

[0023] furthermore, said functional ultrafine particle distribution plasticizer -- particle size 0.2 micrometers Following 0.001 mum the above functional ultrafine particle -- the inside of a plasticizer -- less than [20.0wt%] -- the manufacture approach of the laminated glass which is characterized by being the thing which it makes it come to distribute and which was mentioned above.

[0024] Moreover, said particle size to the solvent for ethylene-vinylacetate copolymer system resin 0.2 micrometers Following 0.001 After distributing the functional ultrafine particle more than mum homogeneity or in the shape of homogeneity at least, [said interlayer] Suitably, with other additives, make ethylene-vinylacetate copolymer system resin carry out the homogeneity dissolution, carry out mixed kneading, and the solvent concerned is film-ized from the raw material resin for film. The manufacture approach of the laminated glass which is characterized by being ethylene-vinylacetate copolymer system resin dried and obtained by 50 - 110 ** and which was mentioned above.

[0025] Moreover, said particle size at least to the ethylene-vinylacetate copolymer system resin which said interlayer heated beyond the temperature of 55-90 degrees C which is a glass transition point, and softened 0.2 micrometers Following 0.001 The manufacture approach of the laminated glass which is characterized by being ethylene-vinylacetate copolymer system resin which added the functional ultrafine particle more than mum directly, and carried out mixed kneading, and which was obtained from the raw material resin for film which carried out homogeneity distribution and which was mentioned above.

[0026] Said functional ultrafine particle Furthermore, Sn, Ti, Si, Zn, Zr, Fe, aluminum, Cr, Co, Ce, In, nickel, Ag, Cu, Pt, Mn, Ta, The metal of W, V, and Mo, an oxide, a nitride, a sulfide, or Sb and F Each independent object of a dope object, Or the manufacture approach of the laminated glass which is characterized by being the coat object which covered the composite which comes to choose at least two or more sorts from these, the mixture which contains an organic resin object in each independent object or a composite concerned further, or an organic resin object and which was mentioned above.

[0027] Furthermore, the manufacture approach of the laminated glass mentioned above that film-ization of said raw material resin for film is characterized by being based on the mold extrusion method or the calendering roll method of a conventional method. Furthermore, the manufacture approach of the laminated glass mentioned above that said laminated-glass-ized processing is characterized by being based on the autoclave method.

[0028] Furthermore, the manufacture approach of the laminated glass which is characterized by basing said laminated-glass-ized processing on heating for 20 - 30 minutes in the temperature requirement of 80 - 120 ** while carrying out a temperature up from ordinary temperature to 120 ** under reduced pressure and which was mentioned above. Here, as described above, particle size is 0.2 in an interlayer layer. mum Making it come to

distribute the following functional ultrafine particles Solar radiation permeability demonstrating enough the functional characteristic of ultrafine particles, such as heat ray electric shielding engine performance, such as 65 etc.% or less, for example controlling the scatter reflection of a light region In order to secure a super-low haze value, electric-wave permeability ability, and transparency, it is because physical properties, such as an adhesive property, transparency, and endurance, are maintained as a conventional interlayer for laminated glass even if it makes an ultrafine particle contain, and it can usually be made to perform laminated-glass-ized processing in an activity with the usual laminated-glass production line. Particle size is 0.15 micrometers preferably. It is [following] extent and is about 0.10-0.001 more preferably. mum It is extent. In addition, about the range of particle size distribution, it is about 0.03-0.01 micrometers, for example. It is good to be equalized with extent.

[0029] Moreover, having presupposed that the mixed rate of the functional ultrafine particle to an interlayer layer is less than [10.0wt%] The amount in which solar radiation permeability demonstrates enough the functional characteristic of ultrafine particles, such as heat ray electric shielding engine performance, such as 65 etc.% or less, for example is secured controlling the scatter reflection of a light region. Even if it makes it be a super-low haze value, electric-wave permeability ability, and transparency furthermore and moreover makes an ultrafine particle contain, as a conventional interlayer for laminated glass For example, an adhesive property, Physical properties, such as transparency and endurance, are maintained and it is because [according to / the usual laminated-glass production line] it can usually be made to perform laminated-glass-ized processing in an activity. When said particle size has a close relation and comes to exceed 10.0wt%, it is because especially the aperture material for automobiles, of course, stops being able to realize the above-mentioned requirements easily also as structural aperture material gradually. When the light permeability Tv is 35% or more as for structural laminated glass especially, for example, the mixed rate of an inorganic pigment system ultrafine particle is the about [abbreviation 10-0.1 wt%] need. It is about 8-0.05wt% more preferably. as the object for construction -- about 9 -- about -0.01wt% -- mixing desirable as an object for automobiles -- if it carries out comparatively -- about 2.0 -- about -0.01wt% -- more -- desirable -- about 1.5 -0.05wt% -- it is 1.0 - 0.1wt % extent still more preferably. Anyway, the mixed rate (content) is determined on the balance of the engine-performance maintenance as laminated glass, and the functional ability to aim at.

[0030] furthermore, an interlayer -- the polyvinyl-butyral system resin film (PVB system) or ethylene-vinylacetate copolymer system resin film (EVA system) it is -- ** -- since these are the things of versatility as an interlayer for laminated glass, having carried out is desirable, and especially if it becomes the interlayer layer which can adjust the quality as laminated glass for needs, it will not limit. In a concrete target, it is plasticity PVB. [The Sekisui Chemical Co., Ltd. make, the Mitsubishi Monsanto Co. make, etc. EVA [the Du Pont make, the Takeda Chemical Industries, Ltd. make, and DEYURAMIN]], and denaturation EVA They are [the TOSOH CORP. make, Mersen G], etc. In addition, addition combination of an ultraviolet ray absorbent, an anti-oxidant, an antistatic agent, a thermostabilizer, lubricant, a bulking agent, coloring, the adhesion regulator, etc. is carried out suitably.

[0031] In addition, or it piles both up for this interlayer containing an ultrafine particle, and the conventional interlayer as an interlayer, it is good also as what is considered as the configuration of sandwiching this interlayer containing an ultrafine particle by the conventional interlayer.

[0032] A functional ultrafine particle Furthermore, Sn, Ti, Si, Zn, Zr, Fe, aluminum, Cr, Co, Ce, In, nickel, Ag, Cu, Pt, Mn, Ta, The metal of W, V, and Mo, an oxide, a nitride, a sulfide, or Sb and F Each independent object of a dope object, Or the composite which comes to choose at least two or more sorts from these, That or it shall be the coat object which covered the mixture or the organic resin object which contains organic resin in an independent object or a composite concerned further It is for discovering suitably heat insulation property, the ultraviolet-rays electric shielding engine performance, the coloring engine performance, protection-from-light nature, etc. as each independent one or a composite, mixture, and a coat object, and making various functionality and engine performance for which the object for construction and automobiles are asked discover as laminated glass.

[0033] moreover -- as a functional ultrafine particle -- Sn, Ti, Si, Zn, Zr, Fe, aluminum, Cr, Co, Ce, In, nickel, Ag, Cu, Pt, Mn, Ta, W, and V etc. -- others -- various metals, such as Mo. for example, SnO_2, TiO_2, SiO_2, ZrO_2, ZnO, Fe 2O3, aluminum 2O3, FeO, Cr 2O3, Co 2O3, CeO_2 and In 2O3, NiO, MnO, and CuO etc. -- various oxides. for example, TiN and AlN etc. -- a nitride or nitrogen oxides. for example, ZnS etc. -- sulfide.

For example, 9wt%Sb2O3-SnO2 (ATO) The [Sumitomo Osaka Cement make], dope object of F-SnO2 grade. Further for example, they are SnO2-10wt%Sb 2O3 and In2O3-5wt%SnO2 (ITO). They are composites, such as the [MITSUBISHI MATERIALS CORP. make]. a fluororesin, PTFE, RUBURON [Daikin Industries, LTD.], SEFURARURU-BU [Central Glass Co., Ltd.], and low molecular weight TFE etc. -- it mentions -- having -- moreover, ATO ITO It has the requirement as an object for automobiles, and is especially desirable.
[0034] Further, for example Co2O3-aluminum 2O3 (TM [3410], 0.01-0.02 micrometers), TiO2-NiO-Co2O3-ZnO (TM3320, 0.01-0.02 micrometers), Inorganic pigment ultrafine particles, such as] by Dainichiseika Colour & Chemicals Mfg. Co., Ltd., are mentioned, respectively. Fe2O3-ZnO-Cr 2O3 (TM [3210], 0.01-0.02 micrometers) -- [-- Moreover, for example as TiO2 ultrafine particle, it is specifically. IT-S-UD [0.02micrometer,] by the Idemitsu petrochemical company, and UF 01[0.018 mum, Tie Oxide Chemicals], etc., Fe 2O3 As an ultrafine particle, nano tightness [ultrafine particle globular form hematite, 0.06 micrometers It cannot be overemphasized that it can be used without limiting especially if the functional characteristic suitably asked also for the ultrafine particle which] by Showa Denko K.K. etc. is mentioned and is not mentioned concretely if needed can be demonstrated maintaining the quality of laminated glass.
[0035] furthermore, about an organic system ultraviolet ray absorbent or an organic system heat ray absorbent As an organic system ultraviolet ray absorbent, it is 2. - (2'- hydroxy-5'-methylphenyl) Benzotriazol, 2 - (2'-hydroxy [- Buthylphenyl] - 3', 5' - JI and tert) Benzotriazol, 2-(2'-hydroxy-3 '-tert-butyl -5'-methylphenyl)-5 - Chlorobenzo triazole, 2-(2'-hydroxy [- Buthylphenyl] - 3', 5' - JI and tert)-5-chlorobenzo triazole, 2 - (2'-hydroxy [- Amyl phenyl] - 3', 5' - JI and tert) Benzotriazol system derivatives, such as benzotriazol, Moreover, 2, 4-dihydroxy benzophenone, 2-hydroxy - 4 - Methoxybenzophenone, 2-hydroxy - 4 - Octoxybenzophenone, 2-hydroxy - 4 - Dodecyloxy benzophenone, 2 2' - Dihydroxy -4 - Methoxybenzophenone, 2, 2' - Dihydroxy -4, a 4'-dimethoxy benzophenone, 2-hydroxy - 4 - Methoxy -5 - Benzophenone system derivatives, such as a sulfo benzophenone, Moreover, 2-ethylhexyl - 2 - Cyano [- Cyanoacrylate system derivatives, such as 3 and 3'-diphenyl acrylate, etc. are mentioned.] - 3, 3'-diphenyl acrylate, ethyl -2 - Cyano Specifically, it is TINUVIN327 [the Ciba-Geigy make] etc.
[0036] Furthermore as an organic system heat ray absorbent, it is NIR-AM1. As a near infrared ray absorbent, it is SIR-114, SIR-128, SIR-130, SIR-132, SIR-169, SIR-103, PA-1001, and PA-1005 to [imperial chemistry industrial company make] and a thing. The [Mitsui Toatsu Chemicals, Inc. make] etc. is mentioned. Especially the thing that can be used without limiting if it demonstrates maintaining the quality of the laminated glass for which the object for construction and automobiles are asked cannot be overemphasized.
[0037] furthermore, the thing for which the laminated glass which becomes with said configuration carried out can be used as various structural aperture material etc. -- of course -- especially -- as the aperture material for automobiles -- for example, a windshield and rear glass -- it can be used for rear glass with a shade band, side glass, sunroof glass, or other various glass at things.
[0038] Furthermore, the particle of organic resin, such as fluororesins, such as PTFE, silicon resin, and silicone rubber, is mentioned, and these are PVB. It is used in order to reduce the bond strength of the film and transparence plates, such as glass. namely, ATO and ITO etc. -- a metallic oxide is used for the same purpose as the coat object which covered organic resin, such as primer spreading on said glass front face, said fluororesin, silicon resin, and SHIRIKO - NGOMU, in order to lower a pan mel value suitably, to adjust it since what gives the bond strength beyond specification may happen, and to lower in a value of standard.
[0039] Generally moreover, as sheet resistance of glass with a glass antenna for example, in case it is the resistance more than 20Kohm/opening and especially an antenna is contacted It is desirable that it is the high resistance 10M omega / more than opening. In the sheet resistance of under 10M omega / opening It is what cannot compare with the electric-wave permeability of the glass plate before making it a layered product, and cannot be stored in the fluctuation difference within 1dB sufficiently stably certainly (as an absolute value). In order [sufficiently stable] to consider as the inside of the fluctuation difference within a less than 1dB fluctuation difference (for example, less than 0.8 dB) certainly, more than 15M omega / opening As sheet resistance of the desirable layered product which furthermore satisfies enough electric-wave permeability ability, an optical property, and physical chemical property, it is the range of 10G omega / below opening extent more than 20M omega / opening. As more desirable sheet resistance, it is the range of 10G omega / below opening extent more than 22M omega / opening.
[0040] this glass plate-like object and said layered product which has almost equivalent electric-wave

permeability ability -- especially -- an optical property top -- skillful -- mutual -- twining -- the synergistic effect -- bringing -- **** -- it considers as what [equipped with the optical function which was markedly alike and was excellent in making it like as well as having raised electric-wave permeability ability and the heat ray electric shielding engine performance] that stood high and is the optimal as a windowpane for automobiles especially.

[0041] that is, electric-wave permeability ability being brought close to said glass plate-like object infinite, and it supposing that it is almost equivalent as a windowpane for automobiles, and solar radiation permeability looking like [65% or less] the heat ray electric shielding engine performance markedly, raising it, and amenity in having improved further The fluoroscopy nature which made the light permeability which an operator, a passenger, etc. need in insurance superiors 65% or more, For example, light permeability secures 70 etc.% or more etc., and can make it possible to also clear a regulation top. And a light reflection factor required for prevention of the fluoroscopy nature fall in an operator, a passenger, etc., misconception, or fatigue of an eye can be made to reduce further from the conventional value, and it becomes the optimal electric-wave transparency mold heat ray ultraviolet-rays electric shielding laminated glass. For 68 - 70% or more, and a light reflection factor, moreover, solar radiation permeability is [light permeability / 60% or less and excitation purity] 15 - 10% or less 14% or less preferably as an object for automobiles, and, for 30% or more and a light reflection factor, moreover, solar radiation permeability is [light permeability / 65% or less and excitation purity] 20% or less 20% or less preferably as an object for construction.

[0042] Further again the laminated glass of said electric-wave transparency mold heat ray ultraviolet-rays electric shielding For example, a perimeter part or a little larger part than the feeding point section is removed from a periphery edge by a certain width of face within the black frame of the periphery of a glass plate-like object. or the part which moreover really casts or post-installs a mall (frame) like this feeding point section -- removing -- further -- this antenna -- a conductor -- it cannot be overemphasized that the configuration, such as adopting the functional interlayer which contains an ultrafine particle except for all or a part of parts, can be made free suitably.

[0043] An interlayer has the heat ray electric shielding engine performance, and the glass which can be prevented from discovering more certainly electromagnetic interferences, such as a ghost phenomenon in the radio disturbance or TV image in broadcast of AM electric wave, FM electric wave, etc., etc. by being the semi-conductor film thru/or an insulator layer, and a high value about sheet resistance, and has sufficient electric-wave permeability ability can be obtained further again, and it can consider as an environment-friendly thing. Moreover, when the direct laminating of the film which has said heat ray electric shielding engine performance of high resistance is carried out to a glass antenna element for example, it can be said that it was made not to affect electric-wave receiving performance degradation.

[0044] Moreover, as described above, it is glass with covering film, such as glass, the primer, the various functional film, etc. which is similar to a transparent clearance thru/or colored glass, tempered glass, or it as a glass plate-like object with minerals glass, organic glass or these compound glass, and the minerals manufactured especially with the so-called float glass process, and it is Green system glass and bronze system glass preferably, and can be adopted as gray system glass, blue system glass, etc. further, for example. Moreover, things which can be further used as various sheet glass products, such as a plate or a bending plate, such as multiple glass besides laminated glass and bilayer glass, cannot be overemphasized. Moreover, as board thickness, it is about 1.0mm. It is about 12mm or less more than extent, and is about 2.0mm as an object for construction. About 10mm or less is desirable more than extent, and it is about 1.5mm as an object for automobiles. It is about 3.0mm more than extent. Below extent is desirable and it is about 2.0mm more preferably. It is about 2.5mm more than extent. It is glass below extent.

[0045] furthermore, PVB A system or EVA the system resin film -- particle size -- 0.2 mum Make it distribute and it considers as a functional ultrafine particle distribution plasticizer. the following functional ultrafine particles -- the inside of a plasticizer -- less than [80.0wt%] -- Subsequently, it is PVB about this functional ultrafine particle distribution plasticizer. A system or EVA In a system resin solution PVB A system or EVA Distributed addition is carried out at least. system resin -- receiving -- a functional ultrafine particle distribution plasticizer -- less than [50wt%] -- That we decided to have added other additives suitably, to have carried out mixed kneading, and to have obtained from the raw material resin for film It is easy to distribute the direction which makes said functional ultrafine particle distribute in a plasticizer solution. Particle size is 0.2. mum

Distribution of the following functional ultrafine particles can be equalized enough. It is because transparency is acquired and is because distribution will become difficult and equalization will become becoming is easy to be trustworthy less gradually, if the amount of mixing exceeds 80.0wt(s)%. desirable -- or less about 20.0wt% -- more -- desirable -- or less about 10.0wt% -- still more preferably, it is extent more than below 5.0wt(s) %0.5wt %, and even if too few, said effectiveness is lost.

[0046] moreover, PVB A system or EVA if distributed addition of a functional ultrafine particle distribution plasticizer exceeds 50wt(s)% to system resin -- PVB A system or EVA since it is easy to come to cause trouble to the engine performance as an interlayer of not only distribution in system resin but laminated glass -- it is -- desirable -- or less about 45wt% -- it is beyond about or less about [40wt%] 10wt% more preferably. Moreover, the usual mixer, a Banbury mixer and the Brabender mixer, a kneader, etc. are used for mixed kneading.

[0047] As a plasticizer, further again, for example Dioctyl phthalate (DOP), Di-isodecyl phthalate (DIDP), di-tridecyl phthalate (DTDP), butyl benzyl phthalate (BBP) etc. -- phthalic ester and tricresyl phosphate (TCP) -- trioctylphosphate (TOP) etc. -- phosphoric ester and tributyl citrate -- methyl acetyl triricinolate (MAR) etc. -- fatty-acid-ester and triethylene glycol G 2- Such mixture, such as polyether ester, such as ethyl butyrate (3GH) and tetraethylene glycol dihexanol, is mentioned.

[0048] Furthermore, said PVB As a solvent which dissolves system resin, ethyl alcohol, n-propyl alcohol, isopropyl alcohol, n-butyl alcohol, a methylene chloride, etc. are mentioned, for example. It is said EVA further again. As a solvent which dissolves system resin, toluene, a xylene, a methylene chloride, etc. are mentioned, for example.

[0049] Furthermore, as film-izing of said raw material resin for film, it is a mold extrusion method or the calendering roll method of a conventional method etc. as the thickness of an interlayer -- about 0.2-1.2mm extent -- desirable -- about 0.3-0.9mm It is extent.

[0050] Furthermore, as said laminated-glass-ized processing, while carrying out a temperature up from ordinary temperature to 120 ** under the autoclave method and reduced pressure, it is heating for 20 - 30 minutes etc. in the temperature requirement of 80 - 120 **, and the crimp of uniform irregularity is prepared in a film front face. In addition, it cannot be overemphasized that various simple laminated-glass-ized processings are applicable suitably with a case.

[0051]

[Function] The laminated glass of this invention as mentioned above Coloring, a heat ray and ultraviolet-rays cutoff film, The particle size which has various kinds of functional ability, such as electric-wave transparency, is 0.2. mum By having considered as the laminated glass which becomes by doubling that it is also in the resin interlayer layer which carries out distributed content of the ultrafine particle which is the following suitably, and processing, and its manufacture approach Without affecting the interlayer layer for laminated glass currently used from the former Functional characteristics, such as heat insulation property, ultraviolet-rays cutoff engine performance, and electric-wave permeability ability, are given, moreover, control and the haze value of the color tone of a clearance thru/or coloring glare with reservation and reflexibility of the fluoroscopy nature excellent very low, and prevention of admiration etc. is brought about with sufficient balance. **, For example, each trial of JIS R 3212 concerning the safety glass for automobiles etc. is cleared, Can acquire the quality which is not different from conventional laminated glass, and neither special component presentation glass nor special surface treatment glass is needed. And it remains as it is, a laminated-glass production line present in use can be performed by laminated-glass-ized processing and the activity, easy moreover, it can respond to the magnitude and the gestalt of glass freely cheaply, and laminated glass can be obtained.

[0052] As a result, while having the ultraviolet-rays cutoff which becomes kind to solar radiation permeability, a great environment, and a great man who the air conditioning effectiveness is heightened [man] and make amenity improve It shall have visible-ray permeability broad from a comparatively high thing to a low thing. Radio disturbance in broadcast of AM electric wave, an FM electric-wave TV electric-wave band, etc. can be reduced. Television for vehicles since it is the electric-wave transparency engine performance of the usual float glass average, Without reducing the receiving engine performance of the glass antenna for radio, a cellular phone, etc. Or can reduce electromagnetic interferences, such as a ghost phenomenon, and the original glass antenna engine performance is demonstrated. Colored [colorlessness to] and the various color tones which can secure vehicle inside and outside and a comfortable environment, and need electric-wave permeability ability,

Or it becomes electric-wave transparency mold heat ray ultraviolet-rays electric shielding glass usable as glass, glass and glass, and laminated glass, such as a synthetic-resin plate and a bilayer, etc. The aperture material for automobiles, for example, a front window, of course especially as structural aperture material In a rear window, a side window or a sunroof, a shade band, etc. It can apply also to the glass for windshields enough especially, and the aperture material for airplanes etc. can be applied broadly, and the laminated glass which has the useful functionality used as the optimal thing for the latest needs, and its manufacture approach are offered.
[0053]
[Example] Hereafter, an example explains this invention concretely. However, this invention is not limited to the starting example.
[0054] Example 120wt%ATO (conductive antimony content stannic acid ghost) Ultrafine particle (particle size of 0.02 micrometers following) distribution content DOP (dioctyl phthalate) 10 g and usual It is PVB (polyvinyl butyral) about DOP 130g. Resin It added to 485g, and with other ultraviolet ray absorbents etc., it grade-scoured for about 15 minutes at about 70 degrees C by the mixer of 3 rolls, and mixed. It is the thickness of about 0.8mm before and after 190 ** with a die pressing appearance machine about the obtained raw material resin for film production. It film-ized to extent and rolled round on the roll. In addition, the crimp of uniform irregularity was prepared in the film front face.
[0055] next, the magnitude 300 [about] -- about 2.3mm in mmx300mm and thickness Clear glass substrate (floor line2.3) the interlayer which prepared two sheets, and cut out and prepared said film in the same magnitude as this substrate -- this -- it inserted between two clear glass substrates, and considered as the layered product.
[0056] Subsequently, degassing reduced pressure of the inside of a bag was carried out, this layered product was put into the vacuum bag made of rubber, after holding about about 20 to 30 minutes with about 80 - 110 ** extent, it once carried out by ordinary temperature, and the layered product taken out from the bag was put into autoclave equipment, the grade carried out pressurization heating for about 20 - 40 minutes with the pressure of about 10-14kg/cm2, temperature 110 [about] - 140 ** extent, and laminated-glass-ized processing was carried out.
[0057] Following measurement and evaluation were performed about the obtained laminated glass.
[Optical property]: The permeability of a between with a wavelength of 340-1800nm was measured with the spectrophotometer (340 the account of type **, Hitachi make), and the light permeability Tv (380-780nm), the solar radiation permeability Ts (340-1800nm), excitation purity, a color tone, etc. were searched for(%) by JIS Z8722 and JIS R3106, or JIS Z 8701.
[Whenever [cloudy]]: Haze value H JIS K6714 It was based and asked by carrying out. As an object for construction, 1% or less was considered as success as an object for automobiles 3% or less.
[Electric-wave permeability] :KEC By method measurement (electric-field shielding-effect measuring instrument), it is 3mm of the usual board thickness about the reflection loss value (dB) of the range of 10-1000MHz of electric waves. A clear glass (floor line3t) veneer article and contrast. The absolute value (**dB) of the difference considered less than 2dB as success.
[Adhesive property]: It is 16**4 at the temperature of -18**0.6 **. Time amount neglect is carried out and it is interlayer exposure extent in exfoliation of the glass after adjustment and in hammer **. Few things were considered as success.
[Thermal resistance]: It is 2 at the boiling underwater of 100 **. After carrying out time amount extent boiling, except for 10mm of circumferences, the thing without abnormalities, such as a crack of generating of the bubble in the remaining part, cloudy weather, and glass, was considered as success.
[Moisture resistance]: After putting for two weeks into adjustment of 50**2 ** and relative humidity 95**4 %, the thing without abnormalities, such as a crack of generating of a bubble, cloudy weather, and glass, was considered as success.
[Electrical characteristics]: Measure with the Mitsubishi Petrochemical tabulation side quantity ohm-meter (HIRESTA HT-210).
[0058] (Sheet resistance) . (M omega/opening). It passes more than 10M omega / opening.
[-- in addition -- fundamental -- the term of JIS R 3212 grade safety glass, especially laminated glass -- conformity.]
The solar radiation permeability Ts about 76.8% Consequently, about 58.6%, [the light permeability Tv] The

neutral color tone of a gray system with excitation purity Pe light at 0.7 % extent, There is also no flash by reflection and the haze value H serves as abbreviation 0.3 % extent. optical properties, such as excellent enough heat ray electric shielding nature, -- markedly -- alike -- high surface resistivity -- usually -- the veneer glass average -- for example, 80MHz (FM radio band) About 520-1630kHz (AM radio band) Electric-wave permeability equivalent to veneer glass is usually shown especially. etc. -- And sufficiently stable adhesive property, outstanding thermal resistance, and outstanding moisture resistance are shown, and all are success. The laminated glass which is not different from usual laminated glass can be obtained, with the outstanding amenity, it is kind to an operator, a passenger, or an environment, safety is high, moreover, AM band is begun, and reception can do various electric waves comfortably. a structural windowpane is natural -- the windowpane for automobiles -- especially -- an antenna -- it was what can adopt enough also to a conductor, simultaneously the windowpane for automobiles which it has, and can reply to expectation enough.

[0059] In addition, when various properties, such as weatherability (change should not have about 1000 hour:light permeability mostly at an example and a sunshine weather meter), were otherwise evaluated, it was what all pass.

[0060] example 220wt%ATO (conductive antimony content stannic acid ghost) Ultrafine particle (particle size of 0.02 micrometers following) distribution content 3GH (triethylene glycol-G 2- ethyl butyrate) It is PVB (polyvinyl butyral) about 10 g and the usual 3GH 130 g. Resin It adds to 485g. Furthermore, it is TOSUPA-RU 120 (Toshiba Silicone) as an adhesion regulator. 5g added, and with other ultraviolet ray absorbents etc., it grade-scoured for about 15 minutes at about 70 degrees C by the mixer of 3 rolls, and mixed. It is the thickness of about 0.8mm before and after 190 ** with a die pressing appearance machine about the obtained raw material resin for film production. Thickness of about 0.8mm which film-ized to extent, rolled round on the roll, and prepared the crimp of uniform irregularity in the front face like the example 1 The interlayer of extent was obtained.

[0061] Next, about 2.0mm in magnitude abbreviation 300mmx300mm and thickness Clear glass substrate (floor line2) It used and considered as the layered product like the example 1. Subsequently, laminated-glass-ized processing was carried out like the example 1.

[0062] The obtained laminated glass was an expected thing which shows each physical properties, such as an optical property Tv was excellent 76.5% and Ts was excellent in H of an optical property like the examples 1, such as 0.4 %, 58.5% and electric-wave permeability, and quality, with sufficient balance.

[0063] Example 320wt%ITO (conductive tin content indium oxide) Ultrafine particle (particle-size 0.1 mum following) distribution content BBP (butyl benzyl phthalate) 10 g and usual It is PVB about BBP90g. Resin Thickness of about 0.8mm which added to 323g and prepared the crimp of uniform irregularity in the front face like the example 1 The interlayer of extent was obtained.

[0064] Next, about 2.0mm in magnitude abbreviation 300mmx300mm and thickness Clear glass substrate (floor line2) It used and considered as the layered product like the example 1. Subsequently, laminated-glass-ized processing was carried out like the example 1.

[0065] The obtained laminated glass was an expected thing which shows each physical properties, such as an optical property Tv was excellent 76.3% and Ts was excellent in H of an optical property like the examples 1, such as 0.4 %, 51.5% and electric-wave permeability, and quality, with sufficient balance. Moreover, the pan mel value was seven to about eight, and was a thing suitable for structural laminated glass.

[0066] Example 420wt%ITO (conductive tin content indium oxide) Ultrafine particle (particle-size 0.1 mum following) distribution content BBP (butyl benzyl phthalate) 10 g and usual It is PVB about BBP90g. Resin It adds to 323g. Furthermore, it is TOSUPA-RU 120 (Toshiba Silicone) as an adhesion regulator. Thickness of about 0.8mm which added 5g and prepared the crimp of uniform irregularity in the front face like the example 1 The interlayer of extent was obtained.

[0067] Next, about 2.0mm in magnitude abbreviation 300mmx300mm and thickness Clear glass substrate (floor line2) It used and considered as the layered product like the example 1. Subsequently, laminated-glass-ized processing was carried out like the example 1.

[0068] The obtained laminated glass was an expected thing which shows each physical properties, such as an optical property Tv was excellent 76.2% and Ts was excellent in H of an optical property like the examples 1, such as 0.4 %, 51.6% and electric-wave permeability, and quality, with sufficient balance. Moreover, the pan mel value was three to about four, and was what is suitable as laminated glass for automobiles.

[0069] Thickness of about 0.8mm which added 10g of organic system heat ray absorbents further, and established the crimp of surface homogeneity irregularity as well as an example 1 to the component and amount of example 5 example 3 The interlayer of extent was obtained.
[0070] Next, the same clear glass substrate as an example 2 (floor line2) It used, and considered as the layered product like the example 1, and, subsequently laminated-glass-ized processing was carried out like the example 1. The obtained laminated glass was an expected thing heat insulation property indicates each physical properties, such as an optical property which was excellent and was excellent in others like the example 1 and electric-wave permeability, and quality, to be with sufficient balance to things from an example 1 although Ts falls [Tv] and light permeability falls [H] a little 32.8% 64.3% in 0.4 % etc.
[0071] Example 620wt%ITO Ultrafine particle (particle-size 0.1 mum following) distribution content It is PVB about DIDP(di-isodecyl phthalate)7g and the usual DIDP95g. Resin Thickness of about 0.8mm which added to 323g and established the crimp of surface homogeneity irregularity as well as an example 1 The interlayer of extent was obtained.
[0072] Next, one in the clear glass of the same magnitude and thickness was changed and used for the Green glass substrate (NFL2), and it considered as the layered product like the example 1. Subsequently, laminated-glass-ized processing was carried out like the example 1.
[0073] The obtained laminated glass was an expected thing 73.3% and Ts indicate [Tv] each physical properties, such as 42.0%, an optical property which H excelled the examples 1, such as 0.2 %, in heat insulation property considerably, and also was excelled like the example 1 and electric-wave permeability, and quality, to be with sufficient balance.
[0074] Example 720wt%ITO Ultrafine particle (particle-size 0.1 mum following) distribution content It is PVB about DIDP(di-isodecyl phthalate)7g and the usual DIDP95g. Resin It adds to 323g and is TOSUPA-RU 120 (Toshiba Silicone) as an adhesion regulator further. Thickness of about 0.8mm which added 5g and established the crimp of surface homogeneity irregularity as well as an example 1 The interlayer of extent was obtained.
[0075] Next, one in the clear glass of the same magnitude and thickness was changed and used for the Green glass substrate (NFL2), and it considered as the layered product like the example 1. Subsequently, laminated-glass-ized processing was carried out like the example 1.
[0076] The obtained laminated glass was an expected thing 73.2% and Ts indicate [Tv] each physical properties, such as 42.1%, an optical property which H excelled the examples 1, such as 0.2 %, in heat insulation property considerably, and also was excelled like the example 1 and electric-wave permeability, and quality, to be with sufficient balance.
[0077] Thickness of about 0.8mm which established the crimp of surface homogeneity irregularity as well as an example 1 in the same component and same amount as example 8 example 6 The interlayer of extent was obtained.
[0078] Next, one in the clear glass of the same magnitude and thickness was changed and used for the blue glass substrate (BFL2), and it considered as the layered product like the example 1. Subsequently, laminated-glass-ized processing was carried out like the example 1.
[0079] For H, Tv of the obtained laminated glass was [Ts] the expected thing heat insulation property indicates each physical properties, such as an optical property which it excelled a little and also was excellent like the example 1 and electric-wave permeability, and quality, to be with sufficient balance from the examples 1, such as 0.2 %, 49.5% 76.0%.
[0080] The same thickness of about 0.8mm as example 9 example 8 One in the clear glass of the same magnitude as a degree and thickness was changed and used for the bronze glass substrate (MFL2) using the interlayer of extent, it considered as the layered product like the example 1, and, subsequently laminated-glass-ized processing was carried out like the example 1.
[0081] The obtained laminated glass was an expected thing which shows each physical properties, such as an optical property Tv was excellent 75.1% and Ts was excellent in H of an optical property like the examples 1, such as 0.2 %, 52.1% and electric-wave permeability, and quality, with sufficient balance.
[0082] The same thickness of about 0.8mm as example 10 example 8 One in the clear glass of the same magnitude as a degree and thickness was changed and used for the gray glass substrate (GFL2) using the interlayer of extent, it considered as the layered product like the example 1, and, subsequently laminated-glass-ized processing was carried out like the example 1.

[0083] The obtained laminated glass was an expected thing which shows each physical properties, such as an optical property Tv was excellent 76.0% and Ts was excellent in H of an optical property like the examples 1, such as 0.2 %, 54.5% and electric-wave permeability, and quality, with sufficient balance.
[0084] Usual [which are an example 1140wt% inorganic pigment ultrafine particle / TM3410 [distribution by Co2O3-aluminum 2O3, particle-size 0.01 - 0.02 mum, and Dainichiseika Colour & Chemicals Mfg. Co., Ltd.] content DOP 20g and usual] It is PVB (polyvinyl butyral) about TCP(tricresyl phosphate)120g. Resin What was added to 480g was scoured like the example 1, and it mixed. It is the thickness of about 0.8mm like an example 1 about this. The interlayer of extent was obtained. Subsequently, laminated-glass-ized processing was carried out like the example 1.
[0085] Tv of the obtained laminated glass was the expected thing it indicates each physical properties, such as an optical property which was excellent almost like the example 1 and electric-wave permeability, and quality, to be with sufficient balance 50.2% if Ts is the color tone of a blue system with vivid Pe of 7.8 % and removes the effect concerning coloring, such as 0.2 %, in H 73.8%.
[0086] Usual [which are an example 1230wt% inorganic pigment ultrafine particle / TiO2-NiO-Co2O3-ZnO, particle-size / of 0.01-0.02 micrometers /, and TM3320 [distribution by Dainichiseika Colour & Chemicals Mfg. Co., Ltd.] content DOP 30g and usual] It is PVB (polyvinyl butyral) about MAR(methyl acetyl triricinolate)100g. Resin What was added to 480g was scoured like the example 1, and it mixed. The interlayer with a thickness of about 0.8mm was obtained for this like the example 1. Subsequently, laminated-glass-ized processing was carried out like the example 1.
[0087] Tv of the obtained laminated glass was the expected thing Ts indicates each physical properties, such as an optical property which was excellent like the example 1 60.2% when Pe is the color tone of 13.8% of skillful Green system and the effect concerning coloring, such as 0.2 %, in H was removed and electric-wave permeability, and quality, to be with sufficient balance 77.8%.
[0088] Usual [which are an example 1330wt% inorganic pigment ultrafine particle / Fe2O3-ZnO-Cr 2O3, particle-size / of 0.01-0.02 micrometers /, and TM3210 [distribution by Dainichiseika Colour & Chemicals Mfg. Co., Ltd.] content DOP 20g and usual] It is PVB (polyvinyl butyral) about 3GH(triethylene glycol G 2-ethyl butyrate)150g. Resin What was added to 480g was scoured like the example 1, and it mixed. It is the thickness of about 0.8mm like an example 1 about this. The interlayer of extent was obtained. Subsequently, laminated-glass-ized processing was carried out like the example 1.
[0089] It was the expected thing shown with sufficient balance [physical properties / each /, such as an optical property which was excellent like the example 1 although influenced concerning coloring / fall / 0.2 % etc. and light permeability / H / although 51.8% and Pe of Tv are / the obtained laminated glass / 67.8% and Ts / a little higher, are the color tone of the skillful Green system, and / a little / and electric-wave permeability, and quality,].
[0090] Example 1420wt%ATO It is PVB (polyvinyl butyral) about 10g of ultrafine particle distribution methyl-ethyl-ketone solutions, and 3GH(triethylene glycol G 2- ethyl butyrate)150g. Resin It added to 490g, and heating kneading lump mixing was carried out for about about 1 hour, decompressing to about 20 mmHg(s) at about 80 degrees C by the mixer of 3 rolls with an adhesion regulator, an ultraviolet ray absorbent, etc. It is the thickness of about 0.8mm like an example 1 about this. The interlayer of extent was obtained. Subsequently, laminated-glass-ized processing was carried out like the example 1.
[0091] The obtained laminated glass was an expected thing which shows each physical properties, such as an optical property Tv was excellent 76.4% and Ts was excellent in H of an optical property like the examples 1, such as 0.4 %, 51.6% and electric-wave permeability, and quality, with sufficient balance.
[0092] PVB which heated to example 15 abbreviation 100 ** extent, and became starch syrup-like (polyvinyl butyral) Resin It is ATO to 490g. 2g of ultrafine particles was added and heating kneading lump mixing was carried out at about 80 degrees C by the mixer of 3 rolls with the ultraviolet ray absorbent etc. for about about 1 hour. It is the thickness of about 0.8mm like an example 1 about this. The interlayer of extent was obtained. Subsequently, laminated-glass-ized processing was carried out like the example 1.
[0093] The obtained laminated glass was an expected thing which shows each physical properties, such as an optical property Tv was excellent 77.5% and Ts was excellent in H of an optical property like the examples 1, such as 0.2 %, 55.7% and electric-wave permeability, and quality, with sufficient balance.
[0094] In addition, it cannot be overemphasized that it carries out like examples 3 and 4 also in examples 1 and

2 and examples 5-15, and it can adjust as the object for construction or an object for automobiles suitably, and can use about a pan mel value.

[0095]

[Effect of the Invention] As mentioned above, this invention is particle size 0.2. mum By having made the following functional ultrafine particles into the laminated glass which carries out distributed content, and its manufacture approach at the interlayer layer Without having big effect on the interlayer layer for laminated glass currently used from the former Functional characteristics, such as heat insulation property, ultraviolet-rays cutoff engine performance, and electric-wave permeability ability, are given, moreover, control and the haze value of the color tone of a clearance thru/or coloring glare with reservation and reflexibility of the fluoroscopy nature excellent very low, and prevention of admiration etc. is brought about with sufficient balance. **, The quality which is not different from conventional laminated glass can be acquired, it remains as it is and a laminated-glass production line present in use can be performed by laminated-glass-ized processing and the activity. Cheaply, easy moreover, it can respond to the magnitude and the gestalt of glass freely, and can carry out. As a result, heighten the air conditioning effectiveness, make amenity improve, and are kind to an environment or a man. Broad fluoroscopy nature can be obtained. AM electric wave, an FM electric-wave TV electric-wave band, etc. Television for [as electric-wave transparency engine performance of the usual float glass average] vehicles, Can secure the glass antenna engine performance for radio, a cellular phone, etc., and the original glass antenna engine performance is demonstrated. A building, and vehicle inside and outside and a comfortable environment can be secured. It becomes electric-wave transparency mold heat ray ultraviolet-rays electric shielding glass usable as laminated glass of colored and various color tones etc. from colorlessness. Of course especially as various structural aperture material, the various aperture material for automobiles, the laminated glass which has the useful functionality which can apply broadly the glass for windshields and the aperture material for airplanes, other industrial glass, etc. especially, and becomes the optimal thing for the latest needs, and its manufacture approach can be offered.

[Translation done.]

PATENT ABSTRACTS OF JAPAN

(11)Publication number : **08-259279**
(43)Date of publication of application : **08.10.1996**

(51)Int.Cl. **C03C 27/12**
B32B 17/10
B60J 1/00
E06B 5/00

(21)Application number : **07-165489**
(22)Date of filing : **30.06.1995**

(71)Applicant : **CENTRAL GLASS CO LTD**
(72)Inventor : **KONDO TAKESHI**

(30)Priority
Priority number : 07 7944 Priority date : **23.01.1995** Priority country : JP

(54) LAMINATED GLASS AND ITS PRODUCTION

(57)Abstract:
PURPOSE: To impart satisfactory heat insulating performance, UV shielding performance, etc., at the time of interposing an intermediate film layer between two transparent glasses to form the laminated glass by dispersing functional superfine particles with the diameter specified or below in the intermediate film layer.
CONSTITUTION: Functional superfine particles (e.g. conductive antimony-contg. tin oxide superfine particles) having ≤0.2μm diameter are dispersed in an intermediate film. A polyvinyl butyral resin film is preferably used as the intermediate film. When the functional superfine particles are dispersed, the particles having ≤0.2μm diameter are uniformly dispersed in a solvent dissolving the polyvinyl butyral resin, and then the solvent is uniformly dissolved in the resin along with an additive such as a plasticizer and kneaded, formed into film and dried. Subsequently, at least two transparent glass sheets are laminated by using the obtained intermediate film and vitrified, and a laminated glass is produced.

LEGAL STATUS

[Date of request for examination]	24.02.1998
[Date of sending the examiner's decision of rejection]	
[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]	
[Date of final disposal for application]	
[Patent number]	3154645
[Date of registration]	02.02.2001
[Number of appeal against examiner's decision of rejection]	
[Date of requesting appeal against examiner's decision of rejection]	
[Date of extinction of right]	

(19)日本国特許庁（ＪＰ）　(12) **公開特許公報**（Ａ）　(11)特許出願公開番号

特開平8－259279

(43)公開日　平成８年(1996)10月８日

(51)Int.Cl.[6]	識別記号	庁内整理番号	ＦＩ		技術表示箇所
Ｃ０３Ｃ　27/12			Ｃ０３Ｃ　27/12	Ｌ	
				Ｎ	
				Ｚ	
Ｂ３２Ｂ　17/10			Ｂ３２Ｂ　17/10		
Ｂ６０Ｊ　1/00			Ｂ６０Ｊ　1/00	Ｈ	

審査請求　未請求　請求項の数25　ＯＬ　（全 11 頁）　最終頁に続く

(21)出願番号　特願平7－165489

(22)出願日　平成７年(1995)６月30日

(31)優先権主張番号　特願平7－7944

(32)優先日　平７(1995)１月23日

(33)優先権主張国　日本（ＪＰ）

(71)出願人　000002200
セントラル硝子株式会社
山口県宇部市大字沖宇部5253番地

(72)発明者　近藤　剛
三重県松阪市大口町1510　セントラル硝子株式会社硝子研究所内

(74)代理人　弁理士　坂本　栄一

(54)【発明の名称】　合せガラス及びその製造方法

(57)【要約】

【目的】　従来の合せガラスと同等の品質を維持しつつ、中間膜層に断熱性能や紫外線遮断性能、電波透過性能等の機能性をもたらしめ、建築用はもちろん、自動車用窓材として各種部署に適用できる合せガラスを得る。

【構成】　少なくとも２枚の透明ガラス板状体の間に中間膜層を有する合せガラスにおいて、該中間膜層の中に粒径が0.2 μm以下の機能性超微粒子を分散せしめてなるものとした合せガラス。

1

【特許請求の範囲】

【請求項１】　少なくとも２枚の透明ガラス板状体の間に中間膜層を有する合せガラスにおいて、該中間膜層の中に粒径が 0.2μm以下の機能性超微粒子を分散せしめてなるものとしたことを特徴とする合せガラス。

【請求項２】　前記中間膜が、ポリビニルブチラール系樹脂膜であることを特徴とする請求項１記載の合せガラス。

【請求項３】　前記中間膜が、エチレン－酢酸ビニル共重合体系樹脂膜であることを特徴とする請求項１記載の合せガラス。

【請求項４】　前記機能性超微粒子の粒径が、0.15～0.001μm であることを特徴とする請求項１乃至３記載の合せガラス。

【請求項５】　前記機能性超微粒子の混合割合が、10.0wt％以下であることを特徴とする請求項１乃至４記載の合せガラス。

【請求項６】　前記機能性超微粒子の混合割合が、 2.0～0.01wt％であることを特徴とする請求項１乃至５記載の合せガラス。

【請求項７】　前記機能性超微粒子が、Sn、Ti、Si、Zn、Zr、Fe、Al、Cr、Co、Ce、In、Ni、Ag、Cu、Pt、Mn、Ta、W 、V 、Moの金属、酸化物、窒化物、硫化物あるいはSbやF のドープ物の各単独物、もしくはこれらの中から少なくとも２種以上を選択してなる複合物、またはさらに当該各単独物もしくは複合物に有機樹脂物を含む混合物または有機樹脂物を被覆した被膜物であることを特徴とする請求項１乃至６記載の合せガラス。

【請求項８】　前記中間膜が、有機系紫外線吸収剤、有機系熱線吸収剤あるいは顔料の各単独もしくはこれらを含有してなることを特徴とする請求項１乃至７記載の合せガラス。

【請求項９】　前記合せガラスが、建築用ガラスであることを特徴とする請求項１乃至８記載の合せガラス。

【請求項10】　前記合せガラスが、自動車用ウインドウガラスであることを特徴とする請求項１乃至８記載の合せガラス。

【請求項11】　少なくとも２枚の透明ガラス板状体の間に中間膜層を有する合せガラスを製造する方法において、粒径が 0.2μm以下の機能性超微粒子を分散せしめた該中間膜を用いて前記少なくとも２枚のガラス板を合せガラス化処理をすることを特徴とする合せガラスの製造方法。

【請求項12】　前記中間膜が、ポリビニルブチラール系樹脂膜であることを特徴とする請求項11記載の合せガラスの製造方法。

【請求項13】　前記ポリビニルブチラール系樹脂膜が、粒径が 0.2μm 以下の機能性超微粒子を可塑剤中に80.0wt％以下分散せしめて機能性超微粒子分散可塑剤とし、次いで該機能性超微粒子分散可塑剤をポリビニルブチラール系樹脂中に、ポリビニルブチラール系樹脂に対し機能性超微粒子分散可塑剤を50wt％以下少なくとも分散添加し、適宜その他の添加剤を加え、混合混練することで機能性超微粒子を均一に分散した膜用原料樹脂から得たことを特徴とする請求項11乃至12記載の合せガラスの製造方法。

2

【請求項14】　前記機能性超微粒子分散可塑剤が、粒径が 0.2μm 以下0.001 μm以上の機能性超微粒子を可塑剤中に20.0wt％以下分散せしめてなるものであることを特徴とする請求項12乃至13記載の合せガラスの製造方法。

【請求項15】　前記中間膜が、ポリビニルブチラール系樹脂を溶解する溶剤に前記粒径が 0.2μm 以下0.001 μm以上の機能性超微粒子を少なくとも均一または均一状に分散した後、当該溶剤を適宜可塑剤ならびにその他の添加剤とともにポリビニルブチラール系樹脂に均一溶解させ混合混練して膜用原料樹脂からフイルム化し、50～110 ℃で乾燥して得たポリビニルブチラール系樹脂膜であることを特徴とする請求項11記載の合せガラスの製造方法。

【請求項16】　前記中間膜が、ガラス転移点である55～90℃の温度以上に加熱して軟化したポリビニルブチラール系樹脂に少なくとも前記粒径が 0.2μm 以下0.001 μm以上の機能性超微粒子を直接添加し混合混練して均一分散した膜用原料樹脂から得たポリビニルブチラール系樹脂膜であることを特徴とする請求項11記載の合せガラスの製造方法。

【請求項17】　前記中間膜が、エチレン－酢酸ビニル共重合体系樹脂であることを特徴とする請求項11記載の合せガラスの製造方法。

【請求項18】　前記エチレン－酢酸ビニル共重合体系樹脂が、粒径が 0.2μm 以下の機能性超微粒子を可塑剤中に80.0wt％以下分散せしめて機能性超微粒子分散可塑剤とし、次いで該機能性超微粒子分散可塑剤をエチレン－酢酸ビニル共重合体系樹脂中に、エチレン－酢酸ビニル共重合体系樹脂に対し機能性超微粒子分散可塑剤を50wt％以下少なくとも添加し、適宜その他の添加剤を加え、混合混練することで機能性超微粒子を均一に分散した膜用原料樹脂から得たことを特徴とする請求項11および17記載の合せガラスの製造方法。

【請求項19】　前記機能性超微粒子分散可塑剤が、粒径が 0.2μm 以下0.001 μm 以上の機能性超微粒子を可塑剤中に20.0wt％以下分散せしめてなるものであることを特徴とする請求項11および17乃至18記載の合せガラスの製造方法。

【請求項20】　前記中間膜が、エチレン－酢酸ビニル共重合体系樹脂用溶剤に前記粒径が 0.2μm 以下0.001 μm以上の機能性超微粒子を少なくとも均一または均一状に分散した後、当該溶剤を適宜その他の添加剤とともにエチレン－酢酸ビニル共重合体系樹脂に均一溶解させ混

合混練して膜用原料樹脂からフイルム化し、50～110 ℃で乾燥して得たエチレン－酢酸ビニル共重合体系樹脂であることを特徴とする請求項11および17記載の合せガラスの製造方法。

【請求項21】　前記中間膜が、ガラス転移点である55～90℃の温度以上に加熱して軟化したエチレン－酢酸ビニル共重合体系樹脂に少なくとも前記粒径が 0.2μm 以下0.001 μm以上の機能性超微粒子を直接添加し混合混練して均一分散した膜用原料樹脂から得たエチレン－酢酸ビニル共重合体系樹脂であることを特徴とする請求項11および17記載の合せガラスの製造方法。

【請求項22】　前記機能性超微粒子が、Sn、Ti、Si、Zn、Zr、Fe、Al、Cr、Co、Ce、In、Ni、Ag、Cu、Pt、Mn、Ta、W 、V 、Moの金属、酸化物、窒化物、硫化物あるいはSbやF のドープ物の各単独物、もしくはこれらの中から少なくとも２種以上を選択してなる複合物、またはさらに当該各単独物もしくは複合物に有機樹脂物を含む混合物または有機樹脂物を被覆した被膜物であることを特徴とする請求項11乃至21記載の合せガラスの製造方法。

【請求項23】　前記膜用原料樹脂の膜化が、常法の型押出し法またはカレンダーロール法によることを特徴とする請求項11乃至17記載の合せガラスの製造方法。

【請求項24】　前記合せガラス化処理が、オートクレーブ法によることを特徴とする請求項11乃至17記載の合せガラスの製造方法。

【請求項25】　前記合せガラス化処理が、減圧下で常温から120 ℃まで昇温する中で80～120 ℃の温度範囲で20～30分間の加熱によることを特徴とする請求項11および18乃至22記載の合せガラスの製造方法。

【発明の詳細な説明】

【０００１】

【産業上の利用分野】本発明は、着色、熱線や紫外線遮断膜、電波透過等各種の機能性超微粒子を適宜有する樹脂中間膜層を用いて合せ処理することでなる合せガラスとその製造方法に関する。

【０００２】冷暖房効果を向上せしめるような優れた日射透過率、環境や人に優しくなる紫外線遮断等を有するとともに、比較的高いものから低いものまで幅広い可視光線透過率を有するものであり、AM電波、FM電波等の放送における受信障害あるいはゴースト現象等の電波障害を低減ができ、電波透過性能を必要とする無色から有色と各種色調の合せガラスとして使用可能な電波透過型熱線紫外線遮蔽ガラス等であって、建築用窓材としてはもちろん、特に自動車用窓材、例えばフロントウインドー、リヤウインドーあるいはサイドウインドーまたはサンルーフ等に、また飛行機用窓材、さらにはその他産業用部材等幅広く適用できる有用な機能性を有する合せガラス及びその製造方法を提供するものである。

【０００３】

【従来技術】近年、建築用ガラスにおけるクリアや着色、断熱や紫外線遮断および電波透過等の機能付与はもちろん、車輌用ガラスにおいても車内に通入する太陽輻射エネルギーを遮蔽し、車内の温度上昇、冷房負荷を低減させる目的から熱線遮蔽ガラス、さらに人的物的両面や環境に優しくするため紫外線遮蔽を付加したものが車輌用に採用されている。また最近は特に該車輌用ガラスにおいて、グリーン色調で充分な可視光透過率を有しながら高熱線紫外線遮蔽性能を持ちかつ各種電波の高透過性能が要求されるようになってきており、なかでも微粒子あるいは超微粒子を合せガラスの中間層に分散したようなものとしては次のようなものが知られている。

【０００４】例えば特開平2-22152 号公報には、短波長光線遮断性合せガラス用中間膜が記載されており、特定された一般式で表されるベンゾトリアゾール誘導体からなる群より選ばれる少なくとも１種の光吸収剤と、少なくとも90重量％が 250～400nmの粒径範囲にある粒径分布の微粒子状無機物質とを含有する可塑化ポリビニルブチラール樹脂よりなり、400nm 以下の波長の光を実質的に遮断し、かつ450nm以上の波長の光を実質的に透過させるものが開示され、光吸収剤の含有量が0.4～6 重量％であり、微粒子状無機物質の含有量が 2～17重量％であることが開示されている。

【０００５】また例えば、特開平4-160041号公報には、自動車用窓ガラスが記載されており、透明板状部材間に平均粒径0.1 μm 以下の超微粒子とガラス成分との混合層を形成してなるものが記載され、透明板状部材間に２超微粒子とガラス成分とを挟み、ガラス成分によって透明板状部材同士を接着すること、あるいは透明板状部材間にプラスチツクの中間層（PVB)を設け、この中間層と各板状部材との間に夫々粒径0.1 μm 以下の超微粒子とガラス成分との混合層を形成してなること、あるいは平均粒径0.3 ～0.5 μm のスペーサ用微粒子を混合層中に混在させること等が開示されている。

【０００６】また例えば、特開平4-261842号公報には、合わせガラスが記載されており、有機ガラスと、透明体と、有機ガラス及び透明体間に配設された中間膜と、を有する合わせガラスであって、中間膜が、ビニルシランをグラフト変性したエチレン・エチルアクリレート共重合樹脂を含有する樹脂組成物にて形成されているものが開示され、樹脂組成物が、ビニルシランをグラフト変性したエチレン・エチルアクリレート共重合樹脂100 重量部と二酸化ケイ素微粒子3 ～30重量部とを含有することが開示されている。

【０００７】

【発明が解決しようとする問題点】前述したような、例えば特開平2-22152 号公報等に記載された短波長光線遮断性合せガラス用中間膜は、ポリビニルブチラール樹脂に添加される少なくとも90重量％が250 ～400nm の粒径範囲にある粒径分布の微粒子状無機物質が光散乱剤とし

て400nm 以下の紫外線部分を散乱させるようにして光吸収剤の選択的吸収を促進し400nm 以下の波長の光を実質的に遮断するとともに、例えば450 ～700nmの波長範囲で光線透過率が70％以上等、450nm 以上の波長の光を実質的に透過させ透明性を保持し、しかも100Wの白色電球像の縁における観察で滲りが無く、黄色味を示す波長420nm における光線透過率も50％以上であって、良好な接着性を示すというものであるが、例えば断熱性能をもたらしめるため、少なくとも90重量％が250 ～400nm の粒径範囲にある粒径分布の断熱性微粒子状無機物質をポリビニルブチラール樹脂に添加した際に、例えば450 ～700nm の波長範囲で光線透過率が70％以上でしかも自動車用窓ガラスとして採用し得るようになることの記載もまた示唆する記載もないものであり、断熱性微粒子状無機物質の粒径が比較的大きいことはもちろんその添加量も例えば2 ～17重量％と多くすることが必要である。

【０００８】また、例えば特開平4-160041号公報に記載された自動車用窓ガラスは、透明板状部材間に平均粒径0.1 μm 以下の超微粒子と有機ケイ素あるいは有機ケイ素化合物のガラス成分との混合層を形成するようにし、合わせガラスのガラス同士あるいはプラスチツクの中間層であるポリビニルブチラール（PVB)とガラスを接合したというものであって、ヒータ用としてのデイフロスタ機能、冷暖房効率アップ用としての赤外線反射機能及び／またはシート抵抗が約500 Ω／口である電磁シールド機能を有することとなるというものであり、PVB やエチレン－酢酸ビニル共重合体系樹脂膜(EVA) 等の中間膜のみで２枚のガラスを接合した通常の合せガラスの構成の中で断熱機能、紫外線遮断機能、電波透過機能あるいは無色乃至着色を同時に発現し得るようなものではないし、また通常の合せガラスと同等の接着力を得ることができるか危惧されるところであり、コスト的にもアップする要因があるものである。

【０００９】また、例えば特開平4-261842号公報に記載された合わせガラスは、有機ガラスを使用するためのものであって、ビニルシランをグラフト変性したエチレン・エチルアクリレート共重合樹脂100 重量部に対し、粒径が0.1 ～400mμのコロイダルシリカや超微粒子シリカ等の二酸化ケイ素微粒子3 ～30重量部とを含有するようにし、粒径を400mμ以下とすることで可視光線の波長(400～780nm)より短いため、中間膜を通過する光の散乱を防ぎ、その中間膜のくもり改善を効果的にしようとするものであるものの、そのくもり度（ヘイズ）はJIS K6714 に基づく測定で４％以下程度であり、必ずしも充分な自動車用窓ガラス、特にフロントガラスとは言い難いものである。

【００１０】

【問題点を解決するための手段】本発明は、従来のこのような点に鑑みてなしたものであり、従来から使用されている合せガラス用中間膜層に影響を与えることなく、中間膜層に機能性超微粒子を適宜分散し含有せしめることで、断熱性能や紫外線遮断性能や電波透過性能等の機能特性を付与し、しかもクリア乃至着色の色調の制御および透視性の確保や反射性とぎらつき感の防止等をバランスよくもたらしめ、従来の合せガラスと変わらない品質を得るようにでき、特殊成分組成ガラスや特殊表面加工ガラスを必要とせず、かつ現在使用中の合せガラス製造ラインをそのままで合せガラス化処理作業で行うことができ、例えばガラスとガラス、ガラスとプラスチック、バイレイヤガラス等を安価にかつ容易にしかもガラスの大きさや形態に自由自在に対応し得て製造でき、建築用窓材はもちろん自動車用窓材、飛行機用窓材、ことに風防用ガラスにも充分適用でき、最近のニーズに最適なものとなる有用な機能的な合せガラスを提供するものである。

【００１１】すなわち、本発明は、少なくとも２枚の透明ガラス板状体の間に中間膜層を有する合せガラスにおいて、該中間膜層の中に粒径が 0.2μm以下の機能性超微粒子を分散せしめてなるものとしたことを特徴とする合せガラス。

【００１２】ならびに、前記中間膜が、ポリビニルブチラール系樹脂膜であることを特徴とする上述した合せガラス。また、前記中間膜が、エチレン－酢酸ビニル共重合体系樹脂膜であることを特徴とする上述した合せガラス。

【００１３】さらに、前記機能性超微粒子の粒径が、0.15～ 0.001μm であることを特徴とする上述した合せガラス。さらに、前記機能性超微粒子の混合割合が、10.0wt％以下であることを特徴とする上述した合せガラス。

【００１４】さらにまた、前記機能性超微粒子の混合割合が、 2.0～0.01wt％であることを特徴とする上述した合せガラス。またさらに、前記機能性超微粒子が、Sn、Ti、Si、Zn、Zr、Fe、Al、Cr、Co、Ce、In、Ni、Ag、Cu、Pt、Mn、Ta、W 、V 、Moの金属、酸化物、窒化物、硫化物あるいはSbやF のドープ物の各単独物もしくはこれらの中から少なくとも２種以上を選択してなる複合物、またはさらに当該各単独物もしくは複合物に有機樹脂物を含む混合物または有機樹脂物を被覆した被膜物であることを特徴とする上述した合せガラス。

【００１５】またさらに、前記中間膜が、有機系紫外線吸収剤、有機系熱線吸収剤あるいは顔料の各単独もしくはこれらを含有してなることを特徴とする上述した合せガラス。

【００１６】またさらに、前記合せガラスが、建築用ガラスであることを特徴とする上述した合せガラス。またさらに、前記合せガラスが、自動車用ウインドウガラスであることを特徴とする上述した合せガラス。

【００１７】ならびに、少なくとも２枚の透明ガラス板状体の間に中間膜層を有する合せガラスを製造する方法において、粒径が 0.2μm以下の機能性超微粒子を分散

せしめた該中間膜を用いて前記少なくとも２枚のガラス板を合せガラス化処理をすることを特徴とする合せガラスの製造方法。

【００１８】また、前記中間膜が、ポリビニルブチラール系樹脂膜であることを特徴とする上述した合せガラスの製造方法。またさらに、前記ポリビニルブチラール系樹脂膜が、粒径が 0.2μm 以下の機能性超微粒子を可塑剤中に80.0wt％以下分散せしめて機能性超微粒子分散可塑剤とし、次いで該機能性超微粒子分散可塑剤をポリビニルブチラール系樹脂中に、ポリビニルブチラール系樹脂に対し機能性超微粒子分散可塑剤を50wt％以下少なくとも分散添加し、適宜その他の添加剤を加え、混合混練することで機能性超微粒子を均一に分散した膜用原料樹脂から得たことを特徴とする上述した合せガラスの製造方法。

【００１９】またさらに、前記機能性超微粒子分散可塑剤が、粒径が 0.2μm 以下0.001 μm以上の機能性超微粒子を可塑剤中に20.0wt％以下分散せしめてなるものであることを特徴とする上述した合せガラスの製造方法。

【００２０】また、前記中間膜が、ポリビニルブチラール系樹脂を溶解する溶剤に前記粒径が 0.2μm 以下0.001 μm以上の機能性超微粒子を少なくとも均一または均一状に分散した後、当該溶剤を適宜可塑剤ならびにその他の添加剤とともにポリビニルブチラール系樹脂に均一溶解させ混合混練して膜用原料樹脂からフイルム化し、50～110 ℃で乾燥して得たポリビニルブチラール系樹脂膜であることを特徴とする上述した合せガラスの製造方法。

【００２１】また、前記中間膜が、ガラス転移点である55～90℃の温度以上に加熱して軟化したポリビニルブチラール系樹脂に少なくとも前記粒径が 0.2μm 以下0.001 μm以上の機能性超微粒子を直接添加し混合混練して均一分散した膜用原料樹脂から得たポリビニルブチラール系樹脂膜であることを特徴とする上述した合せガラスの製造方法。

【００２２】また、前記中間膜が、エチレン－酢酸ビニル共重合体系樹脂であることを特徴とする上述した合せガラスの製造方法。またさらに、前記エチレン－酢酸ビニル共重合体系樹脂が、粒径が 0.2μm 以下の機能性超微粒子を可塑剤溶液中に80.0wt％以下分散せしめて機能性超微粒子分散可塑剤とし、次いで該機能性超微粒子分散可塑剤をエチレン－酢酸ビニル共重合体系樹脂中に、エチレン－酢酸ビニル共重合体系樹脂に対し機能性超微粒子分散可塑剤を50wt％以下少なくとも添加し、適宜その他の添加剤を加え、混合混練することで機能性超微粒子を均一に分散した膜用原料樹脂から得たことを特徴とする上述した合せガラスの製造方法。

【００２３】またさらに、前記機能性超微粒子分散可塑剤が、粒径が 0.2μm 以下0.001 μm 以上の機能性超微粒子を可塑剤中に20.0wt％以下分散せしめてなるものであることを特徴とする上述した合せガラスの製造方法。

【００２４】また、前記中間膜が、エチレン－酢酸ビニル共重合体系樹脂用溶剤に前記粒径が 0.2μm 以下0.001 μm以上の機能性超微粒子を少なくとも均一または均一状に分散した後、当該溶剤を適宜その他の添加剤とともにエチレン－酢酸ビニル共重合体系樹脂に均一溶解させ混合混練して膜用原料樹脂からフイルム化し、50～110 ℃で乾燥して得たエチレン－酢酸ビニル共重合体系樹脂であることを特徴とする上述した合せガラスの製造方法。

【００２５】また、前記中間膜が、ガラス転移点である55～90℃の温度以上に加熱して軟化したエチレン－酢酸ビニル共重合体系樹脂に少なくとも前記粒径が 0.2μm 以下0.001 μm以上の機能性超微粒子を直接添加し混合混練して均一分散した膜用原料樹脂から得たエチレン－酢酸ビニル共重合体系樹脂であることを特徴とする上述した合せガラスの製造方法。

【００２６】さらに、前記機能性超微粒子が、Sn、Ti、Si、Zn、Zr、Fe、Al、Cr、Co、Ce、In、Ni、Ag、Cu、Pt、Mn、Ta、W 、V 、Moの金属、酸化物、窒化物、硫化物あるいはSbやF のドープ物の各単独物、もしくはこれらの中から少なくとも２種以上を選択してなる複合物、またはさらに当該各単独物もしくは複合物に有機樹脂物を含む混合物または有機樹脂物を被覆した被膜物であることを特徴とする上述した合せガラスの製造方法。

【００２７】またさらに、前記膜用原料樹脂の膜化が、常法の型押出し法またはカレンダーロール法によることを特徴とする上述した合せガラスの製造方法。またさらに、前記合せガラス化処理が、オートクレーブ法によることを特徴とする上述した合せガラスの製造方法。

【００２８】またさらに、前記合せガラス化処理が、減圧下で常温から120 ℃まで昇温する中で80～120 ℃の温度範囲で20～30分間の加熱によることを特徴とする上述した合せガラスの製造方法。ここで、前記したように、中間膜層の中に粒径が0.2 μm 以下の機能性超微粒子を分散せしめてなるものとしたのは、可視光域の散乱反射を抑制しながら、例えば日射透過率が65％以下等熱線遮蔽性能等超微粒子の機能特性を充分発揮しつつ、超低ヘーズ値、電波透過性能、透明性を確保するためと、超微粒子を含有せしめても従来の合せガラス用中間膜として例えば接着性、透明性、耐久性等の物性を維持し、通常の合せガラス製造ラインで通常作業で合せガラス化処理ができるようにするためである。好ましくは粒径が0.15 μm 以下程度であり、より好ましくは約0.10～0.001 μm 程度である。なお粒径分布の範囲については、例えば約0.03～0.01μm 程度と均一化されていることがよい。

【００２９】また、中間膜層への機能性超微粒子の混合割合が10.0wt％以下であるとしたのは、可視光域の散乱反射を抑制しながら、例えば日射透過率が65％以下等熱線遮蔽性能等超微粒子の機能特性を充分発揮する量を確

保し、さらに超低ヘーズ値、電波透過性能、透明性であるようにし、しかも超微粒子を含有せしめても従来の合せガラス用中間膜として例えば接着性、透明性、耐久性等の物性を維持し、通常の合せガラス製造ラインによる通常作業で合せガラス化処理ができるようにするためで、前記粒径とも深い関係にあり、10.0wt％を超えるようになると次第に上記要件を特に自動車用窓材はもちろん建築用窓材としても実現し難くなるためである。ことに例えば建築用合せガラス向けとして可視光透過率Tvが35％以上の場合は無機顔料系超微粒子の混合割合が約10～0.1 wt％程度必要であり、建築用としては約９～0.01 wt％程度、より好ましくは８～0.05wt％程度であり、自動車用としては好ましい混合割合としては約2.0 ～0.01 wt％程度、より好ましくは1.5 ～0.05wt％程度、さらに好ましくは1.0 ～0.1wt ％程度である。いずれにしても合せガラスとしての性能保持とめざす機能性能との兼ね合いでその混合割合（含有量）は決定されるものである。

【００３０】さらに、中間膜が、ポリビニルブチラール系樹脂膜(PVB系)、あるいはエチレン－酢酸ビニル共重合体系樹脂膜(EVA系)であるとしたのは、これらが合せガラス用中間膜として汎用性のものであるから好ましく、合せガラスとしての品質をニーズに整合し得るような中間膜層となるものであれば特に限定するものではない。具体的には可塑性PVB〔積水化学工業社製、三菱モンサント社製等〕、EVA〔デュポン社製、武田薬品工業社製、デュラミン〕、変性EVA〔東ソー社製、メルセンG〕等である。なお、紫外線吸収剤、抗酸化剤、帯電防止剤、熱安定剤、滑剤、充填剤、着色、接着調整剤等を適宜添加配合する。

【００３１】なお、中間膜として、本超微粒子入り中間膜と従来の中間膜とを、例えば両者を重ね合わせるあるいは本超微粒子入り中間膜を従来の中間膜でサンドイッチする等の構成とするものとしてもよい。

【００３２】またさらに、機能性超微粒子が、Sn、Ti、Si、Zn、Zr、Fe、Al、Cr、Co、Ce、In、Ni、Ag、Cu、Pt、Mn、Ta、W 、V 、Moの金属、酸化物、窒化物、硫化物あるいはSbやF のドープ物の各単独物、もしくはこれらの中から少なくとも２種以上を選択してなる複合物、またはさらに当該単独物もしくは複合物に有機樹脂を含む混合物または有機樹脂物を被覆した被膜物であるものとしたのは、各単独もしくは複合物、混合物、被膜物として断熱性能、紫外線遮蔽性能、着色性能、遮光性等を適宜発現し、建築用や自動車用に求められる種々の機能性および性能を合せガラスとして発現せしめるためである。

【００３３】また機能性超微粒子としては、例えばSn、Ti、Si、Zn、Zr、Fe、Al、Cr、Co、Ce、In、Ni、Ag、Cu、Pt、Mn、Ta、W 、V 等のほかMoなどの各種金属。例えばSnO_2、TiO_2、SiO_2、ZrO_2、ZnO 、Fe_2O_3 、Al_2O_3 、FeO 、Cr_2O_3 、Co_2O_3 、CeO_2、In_2O_3 、NiO 、MnO 、CuO 等の各種酸化物。例えばTiN 、AlN 等の窒化物、あるいは窒素酸化物。例えばZnS 等の硫化物。例えば9wt%Sb_2O_3-SnO_2(ATO)〔住友大阪セメント社製〕、F-SnO_2等のドープ物。さらに例えばSnO_2-10wt%Sb_2O_3 、In_2O_3-5wt%SnO_2(ITO)〔三菱マテリアル社製〕等の複合物である。フッ素樹脂、PTFE、ルブロン〔ダイキン工業（株）〕、セフラルルーブ〔セントラル硝子（株）〕、低分子量TFE などが挙げられ、またATO やITO は自動車用としてその要件を備え特に好ましいものである。

【００３４】さらに例えばCo_2O_3-Al_2O_3(TM3410、0.01～0.02μm)、TiO_2-NiO-Co_2O_3-ZnO(TM3320 、0.01～0.02μm)、Fe_2O_3-ZnO-Cr_2O_3(TM3210、0.01～0.02μm)〔それぞれ大日精化工業社製〕等の無機顔料超微粒子が挙げられ、また例えば具体的にはTiO_2超微粒子としては IT-S-UD〔0.02μm 、出光石油化学社製〕、UF01〔0.018 μm、タイオキサイド・ケミカルズ社製〕等、Fe_2O_3 超微粒子としてはナノタイト〔超微粒子球形ヘマタイト、0.06μm 、昭和電工社製〕等が挙げられ、具体的に挙げていない超微粒子でも適宜必要に応じて求められる機能特性を合せガラスの品質を維持しつつ発揮することができるものであれば特に限定することなく使用できることは言うまでもない。

【００３５】またさらに、有機系紫外線吸収剤あるいは有機系熱線吸収剤については、有機系紫外線吸収剤としては例えば2-(2'-ヒドロキシ-5'-メチルフェニル) ベンゾトリアゾール、2-(2'-ヒドロキシ-3',5'- ジ・tert-ブチルフェニル) ベンゾトリアゾール、2-(2'-ヒドロキシ-3'-tert- ブチル-5'-メチルフェニル) -5- クロロベンゾトリアゾール、2-(2'-ヒドロキシ-3',5'- ジ・tert - ブチルフェニル)-5-クロロベンゾトリアゾール、2-(2'-ヒドロキシ-3',5'- ジ・tert- アミルフェニル) ベンゾトリアゾール等のベンゾトリアゾール系誘導体、また例えば2,4-ジヒドロキシベンゾフェノン、2-ヒドロキシ-4- メトキシベンゾフェノン、2-ヒドロキシ-4- オクトキシベンゾフェノン、2-ヒドロキシ-4- ドデシルオキシベンゾフェノン、2,2'- ジヒドロキシ-4- メトキシベンゾフェノン、2,2'- ジヒドロキシ-4,4'-ジメトキシベンゾフェノン、2-ヒドロキシ-4- メトキシ-5- スルホベンゾフェノン等のベンゾフェノン系誘導体、また2-エチルヘキシル-2- シアノ-3,3'-ジフェニルアクリレート、エチル-2- シアノ-3,3'-ジフェニルアクリレート等のシアノアクリレート系誘導体などが挙げられる。具体的には例えばTINUVIN327〔チバガイギー社製〕等である。

【００３６】さらに有機系熱線吸収剤としては例えばNIR-AM1 〔帝国化学産業社製〕、ことに近赤外線吸収剤としてはSIR-114 、SIR-128 、SIR-130 、SIR-132 、SIR-169、SIR-103 、PA-1001 、PA-1005 〔三井東圧化学社製〕等が挙げられる。特に建築用や自動車用に求められる合せガラスの品質を維持しつつ発揮するものであれ

ば、限定することなく使用できることは言うまでもない。

【００３７】またさらに、前記した構成でなる合せガラスは種々の建築用窓材等として使用できることはもちろん、特に自動車用窓材として例えばフロントガラス、リアガラスことにシェードバンド付きリアガラス、サイドガラスあるいはサンルーフガラスあるいは他の種々のガラス等に使用できるものである。

【００３８】さらに、PTFEなどのフッ素樹脂、シリコーンレジン、シリコーンゴムなどの有機樹脂の微粒子が挙げられ、これらはPVB 膜とガラスなどの透明板との接着強度を低減するために用いられる。すなわちATO 、ITO などの金属酸化物は規格以上の接着強度を付与するようなことが起こりうるために、パンメル値を適宜下げて調整し規格値内に下げるために、例えば前記ガラス表面へのプライマー塗布、前記フッ素樹脂、シリコーンレジン、シリコ ンゴム等の有機樹脂を被覆した被膜物などと同様の目的で用いる。

【００３９】また、一般にガラスアンテナ付きガラスのシート抵抗値としては、例えば20KΩ／口以上の抵抗値であって、特にアンテナと接触する際には、10M Ω／口以上の高抵抗値であることが好ましく、10M Ω／口未満のシート抵抗値では、積層体にする以前のガラス板の電波透過性に比し充分安定確実に１dB（絶対値として）以内の変動差内に収めることができないものであり、より充分安定確実に１dB以内の変動差内、例えば0.8 dB以内の変動差内とするためには15M Ω／口以上、さらに電波透過性能および光学特性ならびに物理的化学的特性を充分満足する好ましい積層体のシート抵抗値としては20M Ω／口以上10G Ω／口以下程度の範囲であり、より好ましいシート抵抗値としては22M Ω／口以上10G Ω／口以下程度の範囲である。

【００４０】該ガラス板状体とほぼ同等の電波透過性能を有する前記積層体と特に光学特性上で巧みに相互に絡ませ相乗効果をもたらしめるようにすることで、電波透過性能および熱線遮蔽性能を高めたことはもちろん、格段に優れた光学的機能を備える卓越した特に自動車用窓ガラスとして最適なものとしたものである。

【００４１】すなわち、自動車用窓ガラスとして、電波透過性能を前記ガラス板状体に限り無く近づけほぼ同等としかつ熱線遮蔽性能を日射透過率が65％以下と格段に高め居住性をさらに向上したなかで、運転者や搭乗者等が安全上等で必要である可視光透過率を65％以上とした透視性、例えば可視光透過率が70％以上等を確保し法規上もクリアできるようにでき、しかも運転者や搭乗者等における透視性低下、誤認あるいは目の疲労等の防止に必要である可視光反射率を従来の値よりさらに低減せしめることができ、最適な電波透過型熱線紫外線遮蔽合せガラスとなる。自動車用として好ましくは可視光透過率が68～70％以上、可視光反射率が14％以下、しかも日射透過率が60％以下、刺激純度が15～10％以下であり、建築用として好ましくは可視光透過率が30％以上、可視光反射率が20％以下、しかも日射透過率が65％以下、刺激純度が20％以下である。

【００４２】さらにまた、前記電波透過型熱線紫外線遮蔽の合せガラスは、例えばガラス板状体の周辺部の黒枠内で周縁端からある幅で全周部分または給電点部よりやや大きめの部分を除いて、あるいは該給電点部と同様にししかもモール（枠体）を一体成型または後付けする部分を除き、さらには該アンテナ導体部分の全部または一部を除いて超微粒子を含む機能性中間膜を採用する等、その構成は適宜自在になし得ることは言うまでもない。

【００４３】さらにまた、中間膜が熱線遮蔽性能を有してかつシート抵抗値を半導体膜乃至絶縁膜と高い値であることにより、AM電波、FM電波等の放送における受信障害あるいはTV映像でのゴースト現象等の電波障害などをより確実に発現しないようにすることができ、充分な電波透過性能を有するガラスを得て、環境に優しいものとすることができるものである。また例えば、ガラスアンテナ素子に前記高抵抗の熱線遮蔽性能を有する膜を直接積層した場合においても、電波受信性能の低下には影響を及ぼすことがないようにしたと言えるものとなるものである。

【００４４】また、前記したようにガラス板状体としては無機質ガラス、有機ガラスあるいはこれらの複合ガラス、特に所謂フロート法で製造された無機質で透明なクリア乃至着色ガラス、強化ガラスやそれに類するガラス、プライマーや各種機能性膜等被覆膜付きガラスであって、好ましくは例えばグリーン系ガラスやブロンズ系ガラスであり、さらに例えばグレー系ガラスやブルー系ガラス等にも採用可能である。また合せガラスのほか複層ガラス、バイレヤーガラス等、さらに平板あるいは曲げ板等各種板ガラス製品として使用できることは言うまでもない。また板厚としては例えば約1.0mm 程度以上約12mm程度以下であり、建築用としては約2.0mm 程度以上約10mm程度以下が好ましく、自動車用としては約1.5mm 程度以上約3.0mm 程度以下が好ましく、より好ましくは約2.0mm 程度以上約2.5mm 程度以下のガラスである。

【００４５】さらに、PVB 系またはEVA 系樹脂膜が、粒径が0.2 μm 以下の機能性超微粒子を可塑剤中に80.0wt％以下分散せしめて機能性超微粒子分散可塑剤とし、次いで該機能性超微粒子分散可塑剤をPVB 系またはEVA 系樹脂溶液中に、PVB 系またはEVA 系樹脂に対し機能性超微粒子分散可塑剤を50wt％以下少なくとも分散添加し、適宜その他の添加剤を加え、混合混練して膜用原料樹脂から得たこととしたのは、可塑剤溶液中に前記機能性超微粒子を分散せしめる方が分散し易く、粒径が0.2 μm 以下の機能性超微粒子の分散を充分均一化することができ、透明性が得られるためであり、その混合量が80.0wt％を超えると次第に分散が難しくなって均一化が確実で

なくなり易くなるためであり、好ましくは20.0wt％程度以下、より好ましくは10.0wt％程度以下、さらに好ましくは5.0wt ％以下0.5wt ％以上程度であって、少なすぎても前記効果がなくなる。

【００４６】またPVB 系またはEVA 系樹脂に対し機能性超微粒子分散可塑剤の分散添加が50wt％を超えると、PVB 系またはEVA 系樹脂中での分散のみでなく、合せガラスの中間膜としての性能に支障をきたすようになり易いからであり、好ましくは45wt％程度以下、より好ましくは40wt％程度以下10wt％程度以上である。また、混合混練には通常のミキサー、バンバリーミキサーやブラベンダーミキサー、ニーダー等を用いる。

【００４７】さらにまた、可塑剤としては、例えばジオクチルフタレート(DOP) 、ジイソデシルフタレート(DIDP)、ジトリデシルフタレート(DTDP)、ブチルベンジルフタレート(BBP) などのフタル酸エステル、またトリクレシルホスフェート(TCP) 、トリオクチルホスフェート(TOP) などのリン酸エステル、またトリブチルシトレート、メチルアセチルリシノレート(MAR) などの脂肪酸エステル、またトリエチレングリコール・ジ-2- エチルブチレート(3GH) 、テトラエチレングリコール・ジヘキサノールなどのポリエーテルエステルなど、またさらにこれらの混合物が挙げられる。

【００４８】さらに、前記PVB 系樹脂を溶解する溶剤としては、例えばエチルアルコール、n-プロピルアルコール、イソプロピルアルコール、n-ブチルアルコール、メチレンクロライド等が挙げられる。さらにまた、前記EVA 系樹脂を溶解する溶剤としては、例えばトルエン、キシレン、メチレンクロライド等が挙げられる。

【００４９】さらに、前記膜用原料樹脂の膜化としては、常法の型押出し法またはカレンダーロール法等である。中間膜の膜厚としては約0.2 ～1.2mm 程度、好ましくは約0.3 ～0.9mm 程度である。

【００５０】さらに、前記合せガラス化処理としては、オートクレーブ法、減圧下で常温から120 ℃まで昇温する中で80～120 ℃の温度範囲で20～30分間の加熱等であり、膜表面に均一な凹凸のしぼを設ける。なお、場合によって種々の簡易な合せガラス化処理を適宜適用できることは言うまでもない。

【００５１】

【作用】前述したとおり、本発明の合せガラスは、着色、熱線や紫外線遮断膜、電波透過等各種の機能性能を有する粒径が0.2 μm 以下である超微粒子を適宜分散含有せしめた樹脂中間膜層でもって合せ処理することでなる合せガラスとその製造方法としたことにより、従来から使用されている合せガラス用中間膜層に影響を与えることなく、断熱性能や紫外線遮断性能や電波透過性能等の機能特性を付与し、しかもクリア乃至着色の色調の制御およびヘーズ値が極めて低く優れた透視性の確保ならびに反射性とぎらつき感の防止等をバランスよくもたらしめ、例えば自動車用安全ガラスに係わるJIS R 3212の各試験等をクリアする等、従来の合せガラスと変わらない品質を得ることができ、特殊成分組成ガラスや特殊表面加工ガラスを必要とせず、かつ現在使用中の合せガラス製造ラインをそのままで合せガラス化処理と作業で行うことができ、安価にかつ容易にしかもガラスの大きさや形態に自由自在に対応し得て合せガラスを得ることができるものである。

【００５２】ひいては、冷暖房効果を高め居住性を向上せしめるような優れた日射透過率、環境や人に優しくなる紫外線遮断等を有するとともに、比較的高いものから低いものまで幅広い可視光線透過率を有するものとすることができ、AM電波、FM電波TV電波帯等の放送における受信障害などの低減をすることができ、通常のフロートガラス並の電波透過性能であることから、車輌用のテレビ、ラジオ、携帯電話等のためのガラスアンテナの受信性能を低下させることなく、あるいはゴースト現象等の電波障害を低減することができ、本来のガラスアンテナ性能を発揮させ、車輌内外での快適な環境を確保することができることとなり、電波透過性能を必要とする無色から有色と各種色調、はたまたガラスとガラス、ガラスと合成樹脂板、バイレヤー等の合せガラスとして使用可能な電波透過型熱線紫外線遮蔽ガラス等となり、建築用窓材としてはもちろん、特に自動車用窓材、例えばフロントウインドー、リヤウインドーあるいはサイドウインドーまたはサンルーフ、シェードバンド等に、ことに風防用ガラスにも充分適用でき、また飛行機用窓材等幅広く適用でき、最近のニーズに最適なものとなる有用な機能性を有する合せガラス及びその製造方法を提供するものである。

【００５３】

【実施例】以下、実施例により本発明を具体的に説明する。ただし本発明は係る実施例に限定されるものではない。

【００５４】実施例１

20wt％ATO(導電性アンチモン含有錫酸化物) 超微粒子（粒径0.02μm 以下）分散含有DOP(ジオクチルフタレート) 10ｇと通常の DOP 130ｇをPVB(ポリビニルブチラール) 樹脂 485ｇに添加し、他の紫外線吸収剤等とともに３本ロールのミキサーにより約70℃で約15分間程度練り込み混合した。得られた製膜用原料樹脂を型押出機にて190 ℃前後で厚み約0.8mm 程度にフイルム化しロールに巻き取った。なお、フイルム表面には均一な凹凸のしぼを設けた。

【００５５】次に大きさ約300mmx300mm 、厚さ約2.3mm のクリアガラス基板(FL2.3) を２枚用意し、該基板と同じ大きさに前記フイルムを裁断し、調製した中間膜を該２枚のクリアガラス基板の間に挟み積層体とした。

【００５６】次いで該積層体をゴム製の真空袋に入れ、袋内を脱気減圧し、約80～110 ℃程度で約20～30分程度

保持した後一旦常温までにし、袋から取り出した積層体をオートクレーブ装置に入れ、圧力約10～14kg／cm² 、温度約110 ～140 ℃程度で約20～40分間程度の加圧加熱して合せガラス化処理をした。

【００５７】得られた合せガラスについて下記の測定および評価を行った。

〔光学特性〕：分光光度計（340 型自記、日立製作所製）で波長340 ～1800nmの間の透過率を測定し、JIS Z 8722及びJIS R 3106又はJIS Z 8701によって可視光透過率Tv(380～780nm)、日射透過率Ts(340～1800nm) 、刺激純度（％）、色調等を求めた。

〔くもり度〕：ヘーズ値H をJIS K6714 に準拠して行い求めた。建築用としては３％以下、自動車用としては１％以下を合格とした。

〔電波透過性〕：KEC 法測定（電界シールド効果測定器）によって、電波10～1000MHzの範囲の反射損失値（dB）を通常の板厚3mm のクリアガラス(FL3t)単板品と対比。その差の絶対値（△dB）が２dB以内を合格とした。

〔接着性〕： －18±0.6 ℃の温度で16±4 時間放置し調整後、ハンマー打でのガラスの剥離での中間膜露出程度。少ないものを合格とした。

〔耐熱性〕： 100 ℃の煮沸水中にて2 時間程度煮沸した後、周辺10mmを除き、残りの部分での泡の発生、くもり、ガラスのひび割れ等の異常がないものを合格とした。

〔耐湿性〕： 50±2 ℃、相対湿度95±4 ％の調整内に２週間静置した後、泡の発生、くもり、ガラスのひび割れ等の異常がないものを合格とした。

〔電気的特性〕：三菱油化製表面高抵抗計（HIRESTA HT-210）によって測定。

【００５８】（シート抵抗値）（M Ω／口）。10M Ω／口以上合格。

〔なお、基本的にはJIS R 3212等安全ガラス、特に合せガラスの項に準拠。〕

その結果、可視光透過率Tvが約76.8％程度、日射透過率Tsが約58.6％程度、刺激純度Peが0.7 ％程度で淡いグレー系のニュートラル色調、反射によるギラツキもなく、ヘーズ値Hが約0.3 ％程度となり、充分優れた熱線遮蔽性等の光学特性、格段に高い表面抵抗率で通常単板ガラス並み、例えば80MHz(FMラジオ波帯) 、約520 ～1630KHz(AMラジオ波帯) 等特に通常単板ガラスと同等の電波透過性を示し、かつ充分安定な優れた接着性と耐熱性ならびに耐湿性を示しいずれも合格であり、通常の合せガラスと変わらない合せガラスを得ることができ、優れた居住性をもちかつ運転者や搭乗者あるいは環境に優しく安全性が高くしかもAM帯をはじめ各種電波を快適に受信ができ、建築用窓ガラスはもちろん自動車用窓ガラス、ことにアンテナ導体と同時に備える自動車用窓ガラスに対しても充分採用でき、期待に充分答えることができるものであった。

【００５９】なお、他に耐候性（例、サンシヤインウエザーメーターで約1000時間：可視光透過率がほぼ変化がないこと）等の種々の特性をも評価したところ、いずれも合格するものであった。

【００６０】実施例２

20wt％ATO(導電性アンチモン含有錫酸化物) 超微粒子（粒径0.02μm 以下）分散含有3GH(トリエチレングリコール -ジ- 2- エチルブチレート) 10ｇと通常の3GH 130 ｇをPVB(ポリビニルブチラール) 樹脂 485ｇに添加し、さらに接着調整剤としてトスパール120(東芝シリコーン) を５ｇ添加し、他の紫外線吸収剤等とともに３本ロールのミキサーにより約70℃で約15分間程度練り込み混合した。得られた製膜用原料樹脂を型押出機にて190 ℃前後で厚み約0.8mm 程度にフイルム化しロールに巻き取り、実施例１と同様にして表面には均一な凹凸のしぼを設けた厚み約0.8mm 程度の中間膜を得た。

【００６１】次に大きさ約300mmx300mm 、厚さ約2.0mm のクリアガラス基板(FL2) を用いて実施例１と同様にして積層体とした。次いで実施例１と同様にして合せガラス化処理をした。

【００６２】得られた合せガラスは、Tvが76.5％、Tsが58.5％、Hが0.4 ％等実施例１と同様に優れた光学特性ならびに電波透過性、品質等の各物性をバランスよく示す所期のものであった。

【００６３】実施例３

20wt％ITO(導電性錫含有インジウム酸化物) 超微粒子（粒径0.1 μm 以下）分散含有BBP(ブチルベンジルフタレート) 10ｇと通常の BBP90ｇをPVB 樹脂 323ｇに添加し、実施例１と同様にして表面には均一な凹凸のしぼを設けた厚み約0.8mm 程度の中間膜を得た。

【００６４】次に大きさ約300mmx300mm 、厚さ約2.0mm のクリアガラス基板(FL2) を用いて実施例１と同様にして積層体とした。次いで実施例１と同様にして合せガラス化処理をした。

【００６５】得られた合せガラスは、Tvが76.3％、Tsが51.5％、Hが0.4 ％等実施例１と同様に優れた光学特性ならびに電波透過性、品質等の各物性をバランスよく示す所期のものであった。またパンメル値は７～８程度であり、建築用合せガラスに適するものであった。

【００６６】実施例４

20wt％ITO(導電性錫含有インジウム酸化物) 超微粒子（粒径0.1 μm 以下）分散含有BBP(ブチルベンジルフタレート) 10ｇと通常の BBP90ｇをPVB 樹脂 323ｇに添加し、さらに接着調整剤としてトスパール120(東芝シリコーン) を５ｇ添加し、実施例１と同様にして表面には均一な凹凸のしぼを設けた厚み約0.8mm 程度の中間膜を得た。

【００６７】次に大きさ約300mmx300mm 、厚さ約2.0mm のクリアガラス基板(FL2) を用いて実施例１と同様にして積層体とした。次いで実施例１と同様にして合せガラ

ス化処理をした。

【００６８】得られた合せガラスは、Tvが76.2%、Tsが51.6%、Hが0.4 %等実施例１と同様に優れた光学特性ならびに電波透過性、品質等の各物性をバランスよく示す所期のものであった。またパンメル値は３～４程度であり、自動車用合せガラスとして適するものであった。

【００６９】実施例５

実施例３の成分と量に対し、さらに有機系熱線吸収剤10ｇ添加し、実施例１と同様にして表面均一凹凸のしぼを設けた厚み約0.8㎜ 程度の中間膜を得た。

【００７０】次に実施例２と同様のクリアガラス基板(FL2) を用いて実施例１と同様にして積層体とし、次いで実施例１と同様にして合せガラス化処理をした。得られた合せガラスは、Tvが64.3%、Tsが32.8%、Hが0.4 %等、やや可視光透過率が下がるものの実施例１よりことに断熱性能が優れ、他は実施例１と同様に優れた光学特性ならびに電波透過性、品質等の各物性をバランスよく示す所期のものであった。

【００７１】実施例６

20wt%ITO 超微粒子（粒径0.1 μm 以下）分散含有 DIDP(ジイソデシルフタレート)7ｇと通常のDIDP95ｇをPVB樹脂 323ｇに添加し、実施例１と同様にして表面均一凹凸のしぼを設けた厚み約0.8㎜ 程度の中間膜を得た。

【００７２】次に同様の大きさと厚みのクリアガラスのうち１枚をグリーンガラス基板(NFL2)に替えて用い、実施例１と同様にして積層体とした。次いで実施例１と同様にして合せガラス化処理をした。

【００７３】得られた合せガラスは、Tvが73.3%、Tsが42.0%、Hが0.2 %等、実施例１より断熱性能にかなり優れるほか、実施例１と同様に優れた光学特性ならびに電波透過性、品質等の各物性をバランスよく示す所期のものであった。

【００７４】実施例７

20wt%ITO 超微粒子（粒径0.1 μm 以下）分散含有 DIDP(ジイソデシルフタレート)7ｇと通常のDIDP95ｇをPVB樹脂 323ｇに添加し、さらに接着調整剤としてトスパール120(東芝シリコーン) を５ｇ添加し、実施例１と同様にして表面均一凹凸のしぼを設けた厚み約0.8㎜ 程度の中間膜を得た。

【００７５】次に同様の大きさと厚みのクリアガラスのうち１枚をグリーンガラス基板(NFL2)に替えて用い、実施例１と同様にして積層体とした。次いで実施例１と同様にして合せガラス化処理をした。

【００７６】得られた合せガラスは、Tvが73.2%、Tsが42.1%、Hが0.2 %等、実施例１より断熱性能にかなり優れるほか、実施例１と同様に優れた光学特性ならびに電波透過性、品質等の各物性をバランスよく示す所期のものであった。

【００７７】実施例８

実施例６と同様の成分と量で、実施例１と同様にして表面均一凹凸のしぼを設けた厚み約0.8㎜ 程度の中間膜を得た。

【００７８】次に同様の大きさと厚みのクリアガラスのうち１枚をブルーガラス基板(BFL2)に替えて用い、実施例１と同様にして積層体とした。次いで実施例１と同様にして合せガラス化処理をした。

【００７９】得られた合せガラスは、Tvが76.0%、Tsが49.5%、Hが0.2 %等、実施例１より断熱性能がやや優れるほか、実施例１と同様に優れた光学特性ならびに電波透過性、品質等の各物性をバランスよく示す所期のものであった。

【００８０】実施例９実施例８と同様の厚み約0.8㎜ 程度の中間膜を用い、次に同様の大きさと厚みのクリアガラスのうち１枚をブロンズガラス基板(MFL2)に替えて用い、実施例１と同様にして積層体とし、次いで実施例１と同様にして合せガラス化処理をした。

【００８１】得られた合せガラスは、Tvが75.1%、Tsが52.1%、Hが0.2 %等実施例１と同様に優れた光学特性ならびに電波透過性、品質等の各物性をバランスよく示す所期のものであった。

【００８２】実施例10

実施例８と同様の厚み約0.8㎜ 程度の中間膜を用い、次に同様の大きさと厚みのクリアガラスのうち１枚をグレーガラス基板(GFL2)に替えて用い、実施例１と同様にして積層体とし、次いで実施例１と同様にして合せガラス化処理をした。

【００８３】得られた合せガラスは、Tvが76.0%、Tsが54.5%、Hが0.2 %等実施例１と同様に優れた光学特性ならびに電波透過性、品質等の各物性をバランスよく示す所期のものであった。

【００８４】実施例11

40wt%無機顔料超微粒子であるTM3410〔Co_2O_3-Al_2O_3 、粒径0.01～ 0.02 μm、大日精化工業社製〕分散含有DOP 20ｇと通常の TCP(トリクレシルホスフェート)120ｇをPVB(ポリビニルブチラール) 樹脂 480ｇに添加したものを実施例１と同様にして練り込み混合した。これを実施例１と同様にして厚み約0.8㎜ 程度の中間膜を得た。次いで実施例１と同様にして合せガラス化処理をした。

【００８５】得られた合せガラスは、Tvが73.8%、Tsが50.2%、Peが7.8 %の鮮やかなブルー系の色調であって、Hが0.2 %等、着色に係わる影響を除けば、ほぼ実施例１と同様に優れた光学特性ならびに電波透過性、品質等の各物性をバランスよく示す所期のものであった。

【００８６】実施例12

30wt%無機顔料超微粒子であるTM3320〔TiO_2-NiO-Co_2O_3-ZnO、粒径0.01～0.02μm 、大日精化工業社製〕分散含有DOP 30ｇと通常の MAR(メチルアセチルリシノレート)100ｇをPVB(ポリビニルブチラール) 樹脂 480ｇに添加したものを実施例１と同様にして練り込み混合した。これを実施例１と同様にして厚み約0.8㎜程度の中間膜

を得た。次いで実施例１と同様にして合せガラス化処理をした。

【００８７】得られた合せガラスは、Tvが77.8%、Tsが60.2%、Peが13.8%の鮮やかなグリーン系の色調であって、Hが0.2 %等、着色に係わる影響を除けば、実施例１と同様に優れた光学特性ならびに電波透過性、品質等の各物性をバランスよく示す所期のものであった。

【００８８】実施例13

30wt%無機顔料超微粒子であるTM3210〔Fe_2O_3-ZnO-Cr_2O_3、粒径0.01～0.02μm、大日精化工業社製〕分散含有DOP 20gと通常の3GH(トリエチレングリコール・ジ-2-エチルブチレート)150gをPVB(ポリビニルブチラール)樹脂 480gに添加したものを実施例１と同様にして練り込み混合した。これを実施例１と同様にして厚み約0.8mm 程度の中間膜を得た。次いで実施例１と同様にして合せガラス化処理をした。

【００８９】得られた合せガラスは、Tvが67.8%、Tsが51.8%、Peがやや高めではあるが鮮やかなグリーン系の色調であって、Hが0.2 %等、可視光透過率がやや低下するなど着色に係わる影響を受けるものの、実施例１と同様に優れた光学特性ならびに電波透過性、品質等の各物性をバランスよく示す所期のものであった。

【００９０】実施例14

20wt%ATO 超微粒子分散メチルエチルケトン溶液10gと3GH(トリエチレングリコール・ジ-2- エチルブチレート)150gをPVB(ポリビニルブチラール)樹脂 490gに添加し、接着調整剤、紫外線吸収剤などとともに３本ロールのミキサーにより約80℃で約20mmHgに減圧しながら約１時間程度加熱練り込み混合した。これを実施例１と同様にして厚み約0.8mm 程度の中間膜を得た。次いで実施例１と同様にして合せガラス化処理をした。

【００９１】得られた合せガラスは、Tvが76.4%、Tsが51.6%、Hが0.4 %等実施例１と同様に優れた光学特性ならびに電波透過性、品質等の各物性をバランスよく示す所期のものであった。

【００９２】実施例15

約100 ℃程度に加熱して水飴状になったPVB(ポリビニルブチラール)樹脂 490gにATO 超微粒子２gを添加し、紫外線吸収剤などとともに３本ロールのミキサーにより約80℃程度で約１時間程度加熱練り込み混合した。これを実施例１と同様にして厚み約0.8mm 程度の中間膜を得た。次いで実施例１と同様にして合せガラス化処理をした。

【００９３】得られた合せガラスは、Tvが77.5%、Tsが55.7%、Hが0.2 %等実施例１と同様に優れた光学特性ならびに電波透過性、品質等の各物性をバランスよく示す所期のものであった。

【００９４】なお、パンメル値については、実施例１と２ならびに実施例５～15においても実施例３と４のようにして適宜建築用あるいは自動車用として調整して用いることができることは言うまでもない。

【００９５】

【発明の効果】以上前述したように、本発明は粒径0.2μm 以下の機能性超微粒子を中間膜層に分散含有する合せガラス及びその製造方法としたことにより、従来から使用されている合せガラス用中間膜層に大きな影響を与えることなく、断熱性能や紫外線遮断性能や電波透過性能等の機能特性を付与し、しかもクリア乃至着色の色調の制御およびヘーズ値が極めて低く優れた透視性の確保ならびに反射性とぎらつき感の防止等をバランスよくもたらしめ、従来の合せガラスと変わらない品質を得るようにでき、現在使用中の合せガラス製造ラインをそのままで合せガラス化処理と作業で行うことができ、安価にかつ容易にしかもガラスの大きさや形態に自由自在に対応し得て実施でき、ひいては冷暖房効果を高め居住性を向上せしめ、環境や人に優しく、幅広い透視性を得ることができ、AM電波、FM電波TV電波帯等を通常のフロートガラス並の電波透過性能として車輌用のテレビ、ラジオ、携帯電話等のためのガラスアンテナ性能を確保でき、本来のガラスアンテナ性能を発揮させ、建屋や車輌内外での快適な環境を確保することができることとなり、無色から有色と各種色調の合せガラスとして使用可能な電波透過型熱線紫外線遮蔽ガラス等となり、各種建築用窓材としてはもちろん、特に各種自動車用窓材、ことに風防用ガラス、また飛行機用窓材、その他産業用ガラス等幅広く適用でき、最近のニーズに最適なものとなる有用な機能性を有する合せガラス及びその製造方法を提供することができる。

フロントページの続き

(51)Int.Cl.[6]	識別記号	庁内整理番号	FI		技術表示箇所
Ｅ０６Ｂ 5/00			Ｅ０６Ｂ 5/00	Ｚ	